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UMI®
The Processing of Gas Turbine Engine Hot Section Materials Through Directional Solidification

by


A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of

Master of Engineering

Ottawa-Carleton Institute for Mechanical and Aerospace Engineering

Department of Mechanical and Aerospace Engineering
Carleton University
Ottawa, Ontario
Canada

November 1999

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Henry M. J. Saari
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The Processing of Gas Turbine Engine Hot Section
Materials Through Directional Solidification

by

Henry M. J. Saari

in partial fulfillment of the requirements
for the degree of Master of Engineering

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Chair, Department of Mechanical and Aerospace Engineering

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Carleton University
November 1999
Abstract

Directionally solidified superalloy blades are used almost exclusively in the hottest sections of modern aircraft gas turbine engines. The desire to increase engine efficiency is driving research to use lighter materials such as gamma titanium aluminides for some intermediate temperature applications. The development of an appropriate balance of mechanical properties in these intermetallics has proven difficult. However, directional solidification (DS) is a promising method of overcoming this problem through microstructural control.

This thesis describes the development of a Bridgman DS facility for γ-TiAl processing. The facility was commissioned by casting a nickel-base alloy. Temperature gradients in the range of 40 to 78°C/cm were measured for withdrawal rates of $2.82 \times 10^{-3}$ to $4.22 \times 10^{-3}$ cm/s. These conditions produced columnar-grained castings with uniform cellular/dendritic solidification morphology throughout. A composition of Ti-46Al-2Mo-2Nb was chosen for gamma alloy casting trials, which produced bars composed of fully lamellar columnar grains. However, the lamellar orientation did not approach that of the desired target microstructure.
Acknowledgements

I would like to express my sincere gratitude to my advisor, Dr. Jonathan Beddoes, for his continued guidance and advice while working on this extremely exciting project. I would also like to thank my colleague, Dr. Tianyi Cheng, particularly for his excellent work on the gamma alloy metallography. I look forward to continuing this research program with them in the near future. A considerable amount of the project work was performed at the Structures, Materials and Propulsion Laboratory of the Institute for Aerospace Research, National Research Council. I would like to express my appreciation to the staff of M13 and M14 who were involved in the various technical aspects of this project.

I wish to acknowledge Pratt & Whitney Canada, the Natural Sciences and Engineering Research Council of Canada, and the National Research Council Canada for their much-appreciated financial assistance.

For their encouragement and support, special thanks go out to all of my family and friends, especially to all the boys: Tony, Deric, Jeff, Darren, Sid, Karl, and Rick. I would also like to thank Hilary for giving me the added motivation to finish and for the hope of a bright future together.

This thesis is dedicated to my grandfathers, Yrjö and Matti. Both will be greatly missed.
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1 Introduction

1.1 The Gas Turbine Engine

During the last fifty years the continual drive for improvements in efficiency and power output of gas turbine engines has led to ever increasing engine operating temperatures and the use of lighter materials, especially in rotating components. In its simplest form, a gas turbine engine consists of three main components: compressor, combustion chamber, and turbine. A continuous stream of air is compressed by the compressor and enters the combustion chamber where its energy is increased by the combustion of fuel. The hot, high-pressure combustion products expand through the turbine, which drives the compressor, and then through a nozzle which provides the thrust. A simple turbojet, Figure 1-1 [1], illustrates this process. Many variations of this simple scheme are possible. Several independent compressor and turbine stages can be employed and the exhaust gases can be used to drive a separate power turbine. The independent turbine stages can be used to drive a propeller, a separate compressor used for pumping natural gas, or the fan stage of today’s modern high-bypass ratio aircraft engines, shown in Figure 1-2 [1]. Three main factors determine the efficiency and the power output of these engines: turbine inlet temperature (TIT), compressor pressure ratio (CPR), and weight. The influence of TIT and CPR is shown in Figure 1-3 [2]. Generally speaking, the most
efficient operation takes place at high temperatures and compression ratios. Lighter engines result in higher specific thrust and increased payload capacity for aircraft. The use of lighter rotating components is beneficial because higher operating speeds are possible – increasing CPR. These factors have driven research in lightweight, high-temperature materials for the past 50 years and have led to significant advances, especially in turbine blade materials and processing.

1.2 Nickel-base Superalloy Turbine Blades

The rotating turbine blades experience the most arduous conditions of temperature and stress [3]. The combination of high temperatures and stresses leads to creep deformation. Temperature cycling due to changing operating conditions results in thermal fatigue. Aerodynamic forces can induce blade vibrations that cause cyclic stresses, which can lead to high cycle fatigue failures. Oxidation and corrosion are also major concerns. Nickel-base superalloys are used almost exclusively in these applications due to their excellent high temperature strength, creep, and oxidation resistance.

Since the first gas turbine engines, TIT temperatures have more than doubled. Approximately half of this increase can be attributed to alloy development and half to improvements in manufacturing techniques such as blade cooling and directional solidification (DS) and single crystal (SC or SX) technology [4,5]. The trend of increasing temperature capability is illustrated in Figure 1-4 and Figure 1-5. Today’s modern single crystal nickel base superalloy turbine blades incorporating innovative
cooling designs can operate at gas path temperatures well above the melting point of the alloy.

1.3 Why use γ-TiAl Instead?

Gamma titanium aluminides (γ-TiAl) have been the focus of numerous research efforts in the past decade in the aerospace gas turbine engine community [6,7,8,9,10] as candidate materials for use in high temperature structural applications. They will likely be used to replace superalloys and conventional titanium alloys in some intermediate temperature engine component applications such as low-pressure turbine (LPT) and high-pressure compressor (HPC) blades, seal supports, transition duct beams, turbine side plates, and radial diffusers.

There are several reasons why γ-TiAl alloys are suitable replacements for existing alloys in gas turbine engines [9,10,11,12]. The most important is their low density: γ-TiAl alloys have half the weight of superalloys and are about 10-15% lighter than conventional titanium alloys. Their use would allow significant weight savings, yielding increased thrust-to-weight ratios. This is particularly the case in larger high-bypass-ratio engines such as the GE90 where they could, as LPT blade materials, offer a weight savings of up to 45 kg per stage [9], or approximately 200 kg per engine [11]. γ-TiAl alloys have better high-temperature specific strength and stiffness than some superalloys and titanium alloys, as shown in Figure 1-6 and Figure 1-7. In terms of strength, the advantage offered by these alloys seems small. However, in the design of many components, strength is not
the limiting factor. These components are designed according to other factors such as manufacturing tolerances, vibrations, and aerodynamics. One such example is the LPT blades of high-bypass-ratio engines that are made thicker than required for the aforementioned reasons [10]. They are currently cast from nickel-base superalloys, but the operating temperatures and stresses in this part of the engine would allow the use of γ-TiAl alloys instead. The high specific stiffness of these alloys is an advantage in components such as frames, seal supports, and cases where clearances are important. γ-TiAl alloys also have better ignition resistance than conventional titanium alloys, allowing their use in certain applications where the risk of fire, due to blade rubs, restricts the use of conventional titanium alloys [10]. One such example is in the HPC, where the use of titanium blades within a titanium case is avoided and heavier blade materials are used instead. The high resistance of γ-TiAl alloys to ignition would allow the use of γ-TiAl blades within a titanium case, instead of the heavier materials, resulting in weight reduction. The opposite case of titanium blades and a γ-TiAl case would, however, not be acceptable because the ability of γ-TiAl alloys to contain fire is not much greater than that of conventional titanium alloys. Mainly due to the low density of γ-TiAl alloys, but also because of high creep and oxidation resistance, the greatest single payoff area from their use is as replacement materials for current LPT blade materials [10,11].

Unfortunately, a poor balance of mechanical properties resulting from an inverse relationship between tensile properties (strength and ductility) and fracture/creep resistance [6] has limited the use of these alloys. Considerable work has been done in developing new microstructures and in understanding the effects of alloying elements to
overcome this problem. However, it is important to also consider alloy processing in order to improve the balance of mechanical properties of these alloys since \(\gamma\)-TiAl alloy microstructures and, in turn, their mechanical properties depend on processing. One processing route in particular, investment casting, appears to be the most appropriate method of manufacturing \(\gamma\)-TiAl components due to its near-net shape (NNS) processing capability as well as the better mechanical properties of cast material, as compared to wrought or powder metallurgy (PM) processed material [11,12,13]. The NNS capability of investment casting allows lower overall manufacturing costs, due to higher raw material utilization and reduced machining and final finishing operations. It also allows complex component shapes to be produced. \(\gamma\)-TiAl alloys can be readily investment cast using conventional titanium alloy casting methods such as vacuum arc remelting (VAR) [14] or induction skull melting (ISM) [15], with only few process modifications. These methods have been employed in casting \(\gamma\)-TiAl aircraft components that have successfully passed preliminary engine testing. For example, GE Aircraft Engines ran a CF6-80C2 Stage 5 LPT with a full set of \(\gamma\)-TiAl alloy blades in 1993 for 1500 simulated flight cycles with no abnormal blade damage [10,11,12]. Figure 1-8 shows one of these cast blades. Several other engine components were also tested successfully. These initial tests have shown \(\gamma\)-TiAl alloys to be technologically sound materials and have created confidence in the ability to cast commercial products from them. However, for these alloys to be fully accepted and used as high temperature structural materials, further work is required, especially in developing the casting process.
In particular, casting γ-TiAl alloys for blade applications by the DS process used in superalloy casting has generated considerable interest [14,16,17,18,19]. This interest was initially spurred by research with polysynthetically twinned (PST) crystals of binary γ-TiAl alloys [19,20], aimed at gaining a better fundamental knowledge and understanding of the microstructural characteristics and mechanical properties of the fully lamellar microstructure. It is thought that DS processing could be a powerful method of controlling the lamellar structure, thus achieving an adequate balance of mechanical properties and allowing the service stresses and temperatures of γ-TiAl to be increased.

1.4 DS γ-TiAl Project Objectives

To further develop the technology associated with DS processing of γ-TiAl a joint project between Carleton University, the Structures, Materials and Propulsion Laboratory of the National Research Council Canada, and Pratt & Whitney Canada has been initiated. The first goal of this project is the development of equipment and processes for the pilot-scale industrial fabrication of DS γ-TiAl structures, with the ultimate goal being the development of γ-TiAl airfoil-shaped DS structures. This thesis project was undertaken as a part of the overall program, specifically to achieve the first goal of developing the equipment and processes for the pilot-scale industrial fabrication of DS structures. In the following chapter the background knowledge required to support the program is developed through a comprehensive literature review. The review details the current status of DS superalloy and γ-TiAl technology and assesses the technical problems and future work required in making DS γ-TiAl processing a feasible industrial manufacturing
technique. Chapter 3 details the specific objectives of this thesis. The equipment used is described in Chapter 4, while the materials and experimental procedure are presented in Chapters 5 and 6. The last chapter summarizes the work completed, conclusions made, and suggests future research work.
Figure 1-1: Single-spool axial flow turbo-jet engine [1].

Figure 1-2: Triple-spool axial flow front fan turbojet (high by-pass ratio) engine [1].

Figure 1-3: Influence of turbine inlet temperature and compressor pressure ratio on the specific fuel consumption and specific thrust of a gas turbine engine [2].
Figure 1-4: Increase in temperature capability of turbine blade materials for 1000-hour life at a stress of 150 MPa as a function of the year of service introduction [4].

Figure 1-5: The increase of turbine inlet temperature associated with improvements in blade cooling technology [5].
Figure 1-6: Approximate specific tensile and creep strengths of $\gamma$-TiAl, titanium alloys, and nickel-base superalloy 718 as a function of temperature [12].

Figure 1-7: Specific stiffness of Ti4822, Ti-6-4, and alloy 718 as a function of temperature [12].
Figure 1-8: An investment cast turbine blade manufactured for the CF6-80C2 factory engine test [12].
2 Literature Review – DS Processing of Turbine Blades

The following review focuses on the manufacture of gas turbine engine blades through DS processing and consists of four main sections. The first is a review of the history of DS-processed nickel-base superalloy turbine blades, starting with a brief survey of superalloy fundamentals. The basics of the DS process are then reviewed, including heat transfer considerations, plane front solidification, and cellular/dendritic solidification. Following this, some different types of DS furnaces are discussed. The last part of the review covers the present knowledge of DS processing of γ-TiAl alloys for gas turbine applications. It presents some γ-TiAl alloy fundamentals and reviews the current status of DS processing of these alloys.

2.1 History of DS Nickel-base Superalloy Turbine Blades

2.1.1 Nickel-base Superalloy Fundamentals

To fully appreciate the reasons for, and advantages of, DS processing of nickel-base superalloy turbine blades, some understanding of superalloy compositions, microstructures, and processing is required. This section gives a brief overview of these subjects focusing on the nickel-base superalloys (hereafter referred to only as
superalloys). More detailed treatments of nickel-base superalloys and the other superalloy classes are readily available in the literature [21,22,23,24,25].

Superalloys are a family of alloys that were specifically developed for high temperature service. Common characteristics include long-term strength above 650°C and resistance to hot corrosion, oxidation, and erosion. Over the last 50 years these alloys have evolved from a simple nickel-chromium alloy into complex multi-element, multi-phase systems that are capable of operating at temperatures approaching 90% of their absolute melting temperatures. The compositions of representative superalloys developed over the years are shown in Table 2-1.

Nickel-base superalloys consist of a face-centered cubic (FCC) matrix – γ-nickel – with coherent intermetallic precipitates, carbides, borides, and other phases dispersed throughout. Generally, about ten different elements are used for alloying. Some are used for specific purposes (such as chromium used to increase oxidation resistance) and others for multiple uses (such as aluminum that increases oxidation resistance while acting mainly as a strengthener through the formation of precipitates). A list of alloying additions and their purposes is shown in Table 2-2 [26]. The main secondary phases that form are the gamma prime (γ') FCC ordered Ni₃(Al,Ti) intermetallic compound and the carbides MC, M₆C, M₂₃C₆, and M₇C₃, where “M” designates a metal atom. Examples of the types of microstructures possible are shown in Figure 2-1 [23]. Processing and heat treatments play significant roles in forming these phases. The mechanical properties of
superalloys are controlled by three major factors: the amount and morphology of the $\gamma'$ phase, the size and morphology of the grains, and the carbide distribution.

The high strength of superalloys is mainly due to the gamma prime, $\gamma'$, phase that precipitates from solid solution. High strength levels result as $\gamma'$ particles impede dislocation movement, and are directly related to the volume fraction and size of $\gamma'$ particles. Higher volume fractions of $\gamma'$, with an optimum particle size of approximately 0.1 $\mu$m in diameter, lead to higher strength levels, Figure 2-2 [27] and Figure 2-3 [24]. The volume fraction of $\gamma'$ is directly related to the amount of the hardening elements aluminum and titanium present in the alloy – higher aluminum and titanium content leads to higher $\gamma'$ volume fractions. $\gamma'$ particles are formed by heat treatments involving three fundamental steps. The first is solutionizing to dissolve precipitable phases into solid solution. The solid solution is then quenched to form a supersaturated solid solution. An aging treatment is used to precipitate $\gamma'$ out of solution. To obtain the most favourable microstructure, several aging treatments may be necessary.

Processing, which has a direct influence on grain size and morphology, is another important factor [24]. For example, wrought superalloys generally have fine, uniform grain sizes, yielding improved tensile and fatigue properties while cast superalloys have coarser grains that yield better creep and rupture properties. Therefore the end use of a component has direct bearing on what type of processing should be used in its manufacture.
The formation of carbides, in the grains themselves or at grain boundaries, is important as well – especially for improving creep life. Carbides can strengthen the matrix, but contribute only a small amount to the overall strength of nickel superalloys. Their principal role is grain boundary hardening. The presence of discrete globular M23C6 carbides at the grain boundaries enhances creep rupture life by preventing grain boundary sliding, while allowing sufficient ductility in the surrounding grain for stress relaxation to occur – preventing premature failure. This behaviour is especially important for polycrystalline superalloys. However, if carbides form as a continuous grain boundary film or as discontinuous (cellular) structures creep rupture life can be significantly lowered. The different morphologies in which carbides may form are illustrated in Figure 2-4 [28,29].

2.1.2 Wrought and Cast Turbine Blades

The first superalloy turbine blades were developed as wrought alloys [3,4]. Improvements in strength were made by the addition of small amounts of aluminum and titanium, leading to higher γ' volume fractions. The introduction of vacuum induction melting (VIM) was a major breakthrough in this type of alloy development. Larger amounts of aluminum and titanium could be added because melting in vacuum prevented the oxidation of these reactive hardening elements. Increased additions of the hardening elements led to an increase in the γ' solutionizing temperature and a decrease in the incipient melting temperature of the alloy. Consequently, the hot working of these alloys became more difficult as the processing window (the difference between the γ'
solutionizing temperature and the incipient melting temperature of the alloy) became progressively smaller. To allow for practical temperature control during hot working, the size of the processing window was increased by decreasing the chromium content, thus lowering the $\gamma'$ solutionizing temperature. This had an undesirable effect, however, because high chromium content was important for oxidation and corrosion resistance. As alloy development to increase strength continued, progressively smaller processing windows resulted. Forging difficulties were also encountered since better high temperature strength generally leads to poor forgeability. This led to the use of investment casting as an alternative method of manufacturing turbine blades.

Precision vacuum investment casting offered several advantages over wrought processing. Some were due to the casting process itself: its NNS capabilities reduced raw material and machining costs, it was an easy, reliable, and inexpensive method of making complex shaped parts, and it allowed blade designs with complex internal cooling passages (that permitted further increases in temperature capability, Figure 1-5). Cast materials also had coarser grain sizes than fine-grained forgings, and were thus intrinsically stronger at high temperatures. Because forgeability was no longer a concern, alloy microstructures could be specifically tailored (e.g. higher additions of aluminum and titanium) for increased high-temperature strength. Figure 2-5 compares the microstructures of a wrought and a cast superalloy [30]. The increased $\gamma'$ volume fraction is readily apparent. However, as operating temperatures increased it became apparent that the limit of alloy modifications, for increasing material temperature capability, was again being reached. Reduced intermediate temperature creep rupture
ductility was found to be a major problem in cast alloys. The addition of hafnium to alloy compositions to strengthen grain boundaries was one solution to this problem. Greatly increased creep rupture ductility was achieved, Figure 2-6 [3]. Another method involved the elimination of transverse grain boundaries.

2.1.3 DS and SX Turbine Blades

VerSnyder and his colleagues at Pratt & Whitney Aircraft (PWA) developed the DS process to overcome the low creep rupture ductility problem by eliminating transverse grain boundaries [31,32,33,34,35]. It was commonly known that low temperature fracture, in metals, occurred transgranularly (grain boundaries stronger than grains) and high temperature fracture occurred intergranularly (grain boundaries weaker than grains) [36]. It was also known that, at high temperatures, intergranular failure was a result of unfavourable occurrences at the grain boundaries – excessive precipitation, cavitation, and void formation – and was initiated at the grain boundaries that were oriented normal to the applied stress. It was reasoned that the reduction or elimination of these grain boundaries could improve material properties. Some of these improvements are illustrated in Figure 2-7 and Figure 2-8, showing the higher creep rupture ductility and creep life of DS and SX superalloys over conventionally cast superalloys. DS processing also offered additional benefits such as a preferred crystallography and reduced microporosity. The preferred direction of columnar dendrite growth during DS processing yielded a material with a modulus of elasticity 40% lower than polycrystalline material. This resulted in much better resistance to thermal fatigue caused by the differential expansion of material during temperature cycling. Reduced microporosity,
which led to better high cycle fatigue life, was a result of better feeding because of the constant supply of molten alloy at the solidification front.

The main characteristic of DS structures, as opposed to the equiaxed ones found in conventional castings, is that the primary dendrites and grain boundaries are aligned with the main heat transfer, or growth, direction. The heat transfer basics and the furnaces in which these structures are formed are covered in Sections 2.2 and 2.3. For superalloys, like other cubic metals, the preferred growth direction is <100> [37]. Dendrites with this orientation and aligned with the main heat transfer direction outgrow and crowd out less favourably oriented ones. As growth proceeds, favourably oriented dendrites form the columnar grain structure, Figure 2-9 [4]. SX structures are an extension of DS structures where grain boundaries are eliminated completely. The differences between equiaxed, DS, and SX blades are illustrated in Figure 2-10 [38]. SX structures are formed by allowing only a single grain, or crystal, to grow into the component cavity. A seed crystal or a helical grain selector is typically used to accomplish this. In the case of the grain selector, solidification starts as in DS solidification. However, only a few grains can grow into the selector and as solidification proceeds only one grain emerges and fills the component cavity. This process is shown schematically in Figure 2-11 [39]. An etched SX blade is shown in Figure 2-12 [40].

As mentioned above, SX casting was a logical extension of the DS process, eliminating all grain boundaries. However, initial research on DS MAR M200 and DS MAR-M200 + Hf, showed slight, or no, improvements in material properties gained by SX processing.
Later research on DS MAR M200 + Hf showed that by increasing the solutionizing and incipient melting temperatures of the alloy higher $\gamma'$ volume fractions, leading to higher creep strengths, could be obtained [43,44]. This led to the removal of the grain boundary strengthening elements (C, B, Zr, Hf, and Mg) which, in turn, led to an increase of about 100°C in the alloy's incipient melting temperature. A higher incipient melting temperature allowed for a higher solution heat treatment temperature, allowing for the complete solutionization of the $\gamma'$ phase and significantly reduced dendritic segregation.

Based on this research, the development of the first generation SX alloy 454 (PWA 1480) was performed at PWA [45]. Significant increases in creep strength, thermal fatigue life, and oxidation resistance over DS material were achieved in the new alloy (Figure 2-13, Figure 2-14, and Figure 2-15). Because of the absence of any grain boundary strengtheners, the formation of unwanted grain boundaries must, at all costs, be avoided as they could cause catastrophic failures. Unwanted grain boundaries could be formed by freckling (chains of equiaxed grains caused by lower density liquid "jets" rising through the mushy zone during solidification), the formation of equiaxed grains, or recrystallization, during heat treatments, due to mechanical damage caused during stripping and cleaning operations. Process and quality control are important in preventing the formation of unwanted grain boundaries while non-destructive inspection (NDI) techniques are important in detecting them.
Second and third generation SX alloys have attained even better temperature capabilities. Second generation SX alloys such as CMSX-4 [46] and PWA 1484 [47], with 3% rhenium content, have a 30-35°C increase in temperature capability over first generation alloys. Third generation alloys such as CMSX-10 [48], and René N6 [49] have another 30°C improvement. Generally, the trend in these alloy developments was to increase the total refractory metal (W, Ta, Re) content (to increase creep strength) and to increase Al and decrease Ti contents (to increase oxidation resistance). Increases in thermal mechanical fatigue resistance are thought to be due to the increased creep resistance of these alloys.

2.2 The Basics of DS

As mentioned in Section 2.1.1, superalloys are very complex, containing many alloying additions and several different phases. In spite of this they solidify mainly as a single phase – the γ matrix. The γ' phase precipitates at lower temperatures and the other minor phases have little effect on solidification. Therefore the solidification behaviour of superalloys can be described generally by the relationships developed for single-phase solidification. These relationships, as they relate to DS processing, have been reviewed in detail by McLean [4], Flemings [50], and Kurz and Fisher [51]. This section gives a brief review of their work.
2.2.1 Heat Flow

To produce DS structures, where dendrites grow from one end of the casting to the other, two conditions must be met. The first is the establishment of a thermal gradient across a liquid-solid interface. The second is the translation of the gradient to move the liquid-solid interface in a controlled manner. The magnitude and rate of travel of the gradient, as it passes through the casting, controls the resulting microstructure of the solidified material. The methods by which the magnitude and rate of travel of the gradient are controlled are detailed in Section 2.3.

A heat balance equation that relates the heat of fusion to the thermal conductivities of the liquid and solid alloy can be written for a planar liquid-solid interface moving at a velocity, \( V_I \) as follows:

\[
K_S G_S - K_L G_L = \rho_S L_f V_I
\]  
(2.1)

where:

- \( K_S \) = thermal conductivity of solid metal
- \( K_L \) = thermal conductivity of liquid metal
- \( G_S \) = thermal gradient in the solid at the liquid-solid interface
- \( G_L \) = thermal gradient in the liquid at the liquid-solid interface
- \( \rho_S \) = density of the solid metal
- \( L_f \) = latent heat of fusion
- \( V_I \) = velocity of the liquid-solid interface
Equation 2.1 shows that $V_I$ is dependent on the difference between the heat conducted through the solid and the liquid phases, $K_S G_S$ and $K_L G_L$. Therefore, thermal gradients can be controlled independently of $V_I$. This is important for influencing the microscopic solidification morphology. If the melt were undercooled ($G_L < 0$) good crystal growth could not occur as equiaxed grains could nucleate ahead of the liquid-solid interface. Therefore, a maximum liquid-solid interface velocity, $V_{I_{\text{max}}}$, occurs when $G_L = 0$ and it can be increased by increasing $G_S$. When $V_I < V_{I_{\text{max}}}$, $G_S$ and $G_L$ adjust to satisfy Equation 2.1, while at the same time remaining compatible with the temperature gradient imposed by the furnace arrangement. Schematic temperature gradients are shown in Figure 2-16 and show that low $V_I$ results in high $G_L$, an important factor that influences solidification morphology. Increasing the furnace temperature also leads to a high $G_L$ [52].

### 2.2.2 Plane Front Solidification

The solidification of many materials from their melts can be approximated by the assumption of equilibrium at the solid-liquid interface. This means that although there may be large concentration gradients in the solid and the liquid during solidification, the concentration at the liquid-solid interface is given by the equilibrium phase diagram. Solidification also typically occurs over a range of temperatures, rather than at a discrete melting temperature, leading to microsegregation within the alloy. Consider a binary alloy bar of composition $C_0$ solidifying with a planar liquid-solid interface, Figure 2-17. For this general case, the melting temperature decreases with increasing solute concentration. At temperature $T^*$, equilibrium requires the liquid and solid compositions
at the interface to be $C_L^*$ and $C_S^*$, respectively, as fixed by the equilibrium phase
diagram. To relate the liquid and solid compositions at the interface, it is convenient to
define an equilibrium partition ratio, $k$, as $C_S^*/C_L^*$. For the idealized case shown in
Figure 2-17, where liquidus and solidus boundaries are linear, $k$ is a constant. The
compositions of the liquid and the solid away from the interface typically differ from $C_L^*$
and $C_S^*$.

While the assumption of equilibrium at the interface may be valid, solidification is almost
never slow enough to approach true equilibrium conditions. Complete diffusion of solute
in both the liquid and solid states is required for this to occur. It would result in the
solidified bar having a uniform composition of $C_o$ throughout. In practical situations,
however, some casting segregation always occurs because of limited diffusion. It is
usually assumed that no diffusion of solute occurs in the solid and that diffusion is
limited in the liquid. Equilibrium conditions only occur at the liquid-solid interface. This
situation is illustrated in Figure 2-18. When solidification starts, the initial solid forms
with composition $kC_o$ — lower in solute than the initial liquid. The excess solute is
rejected from the interface into the liquid. An enriched solute boundary layer is thus
formed, in the liquid ahead of the solidifying front, due to limited diffusion in the liquid.
During an initial transient period, the composition of the solid increases from $kC_o$ until a
steady state is reached. At steady state the composition of the solid that forms is $C_o$. The
composition of the liquid at the interface is $C_o/k$ and solidification occurs at $T_S$. The
concentration of solute in the liquid in this boundary layer decays from $C_o/k$ to $C_o$. This
steady state holds until the solute boundary layer impinges on the end of the bar, forming

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a final transient of increasing solute concentration in the solid. A bar solidified in this manner will have a uniform composition except at the ends where the initial and final transients occurred.

2.2.3 Cellular/Dendritic Solidification

Cellular or dendritic solidification is a result of instability of the planar liquid-solid interface. As described in the section above, the solute rich boundary layer ahead of the liquid-solid interface causes the instability. It causes a local variation in the liquidus temperature as shown in Figure 2-19. The liquidus temperature is decreased by the boundary layer because it decreases with increasing solute concentration, Figure 2-18 (c). Depending on the actual temperature profile in the liquid, due to the furnace arrangement, two different situations can occur, Figure 2-20. In the first, Figure 2-20 (a), the actual temperature in the liquid is above the liquidus temperature. This is the necessary condition for the plane front to remain stable. If a perturbation forms on the flat interface it will be in a superheated environment, and thus it will re-melt. In the second case, Figure 2-20 (b), the actual temperature in the liquid is below the liquidus temperature, causing interface instability. Now, if a perturbation forms, it will be in a supercooled environment and will grow further into the liquid. This supercooling is termed constitutional supercooling because is it due to a change in composition and not a change in temperature. The condition for stability of the plane front was developed quantitatively by Chalmers and co-workers [53,54] and is given by:

\[
\frac{G_L}{V_l} \geq -\frac{m_L C_0 (1 - k)}{k D_L} \frac{\Delta T}{D_L} \tag{2.2}
\]
where: \( m_L \) = slope of the liquidus boundary

\( D_L \) = diffusion coefficient of solute in the liquid

\( \Delta T \) = melting range of the alloy at the composition \( C_0 \)

Plane front solidification will occur if \( G_l/V_l \) is above a certain critical value, given by Equation 2.2. If it falls below the critical value, due to a drop in gradient and/or an increase in solidification velocity, instabilities develop and a progression of solidification morphologies will occur, Figure 2-21. The effects of increasing solidification velocity, at a constant gradient, on the variation in liquidus temperature ahead of the liquid-solid interface are shown in Figure 2-22. As \( V_l \) increases, constitutional supercooling increases. This is in contrast to the thermal supercooling due to the temperature gradient imposed by the furnace arrangement, which increases with decreasing \( V_l \) (c.f. Figure 2-16).

When the interface first becomes unstable, a cylindrical cellular structure forms. This is mainly a result of solute diffusion, and at slow solidification velocity the cylindrical cells grow normal to the interface regardless of their crystallographic orientation. As the solidification velocity increases, crystallographic effects begin to exert an influence and the cell growth direction deviates toward the preferred crystallographic growth direction - \(<100>\) for cubic materials [37]. Crystallographic effects also affect the cross section of the cells, causing them to become, in cubic materials, flange-shaped or maltese-cross-shaped. With further increases in solidification rate the flanges break down to form secondary and higher order arms. The cells are now termed dendrites. The formation of
the secondary arms is driven by constitutional undercooling as well, as the interface just behind the cell/dendrite tip becomes unstable. This progression of solidification morphologies is illustrated in Figure 2-21, a-e. These morphologies are plotted on a map of \( \log V_1 \) vs. \( \log G_L \) in Figure 2-23 for several DS superalloys grown at various solidification rates and gradients. The different morphologies fall into clear zones on this map.

2.2.3.1 Dendrite Arm Spacing

The most common growth morphologies observed during commercial and laboratory DS processing of superalloys are cellular and dendritic structures [55]. The spacing of the cells/dendrite arms of the microstructure has a large influence on the mechanical properties of the alloy [56,57]. For example, a smaller dendrite arm spacing (DAS) leads to lower segregation in dendrite cores and interdendritic regions, thus benefiting the final mechanical properties. The primary dendrite arm spacing (PDAS), \( \lambda_1 \), is very sensitive to both \( G_L \) and \( V_1 \) and typically spans a range of approximately 20 to 300 \( \mu \text{m} \) in typical commercial and laboratory DS processes [55]. The product of \( G_L \) and \( V_1 \) is termed the cooling rate and influences \( \lambda_1 \) as shown in Figure 2-24 [57]. Higher cooling rates lead to finer PDAS.

The PDAS is the perpendicular distance between cells or dendrite arm branches and can be measured from a transverse section in two ways. The line counting method directly measures the spacing. However, it is subjective because grain boundaries and areas of growth faults tend to be ignored and only the perfectly shaped cells/dendrites are counted.
A more objective and reproducible method is to count the number, \( N \), of cells/dendrites in a given area, \( A \), and using the model of hexagonal or square arrangement of cells/dendrites, obtain an average spacing, \( \lambda_{\text{avg}} \) [58]. For large values of \( N \), both arrangements result in approximately the same equation:

\[
\lambda_{\text{avg}} = \sqrt{A / N}
\]  

(2.3)

The relationship between cell/dendrite spacing and the segregation behaviour on the processing parameters is important from the point of view of modeling the formation of defects such as microporosity, macrosegregation, and freckles [56]. Several models have been proposed to relate \( \lambda_1 \) to \( G_L \), \( V_t \), and the physical properties of the alloy. Most of these models were derived for simple binary alloys but they have been shown to be applicable to complex superalloys as well, as described below. PDAS provides a convenient method to measure the effects of solidification conditions on the solidification morphology [50]. It also provides a convenient way of estimating the temperature gradient in an alloy during solidification when measurement of the actual gradient, with thermocouples placed in the melt, might be too difficult to perform.

Several different functional relationships have been proposed in the literature by which \( G_L \) and \( V_t \) control PDAS. One such relationship was shown in Figure 2-24, which can be expressed by an equation of the form:

\[
\lambda_1 = C(G_L V_t)^p
\]  

(2.4)

However, as demonstrated by Kurz and Fisher [51], \( \lambda_1 \) cannot be predicted by a simple relationship such as Equation 2.4. Their qualitative description of \( \lambda_1 \), based on a very
simplified model, demonstrates that $G_L$ and $V_t$ must have different functional relationships to $\lambda_1$. This is also born out by Hunt's analysis [59]. Using the previous work of Burden and Hunt [60,61], Hunt showed analytically that $\lambda_1$ was proportional to the product of $G_L^{-0.5}$ and $V_t^{-0.25}$ for simple binary alloys. This model has been shown to be valid for superalloys as well. Experimental PDAS for three superalloys is plotted vs. the product $G_L^{-0.5}V_t^{-0.25}$ in Figure 2-25. Those alloys that solidified under conditions to produce a well-defined dendritic structure fall on a common line but alloys that solidified under conditions to produce a planar growth interface did not. It should be noted that two superalloys, IN738LC and MAR M246, seem to fit both of the above models (Figure 2-24 and Figure 2-25). This is most probably due to experimental scatter and the fact that the experimental results are plotted on a log-log scale in Figure 2-24. More recent quantitative comparisons on succino-nitrite-acetone by Trivedi [62] have shown that Hunt's model underestimates the experimentally observed PDAS. The PDAS of PWA1480 measured by Vijayakumar et al. [56] show good agreement with Trivedi's model [62]. This is illustrated in Figure 2-26. The least-squared fit to the experimental data of Vijayakumar et al. is given by the equation:

$$\lambda_1 = (-0.00128 \pm 0.0013) + (0.0728 \pm 0.0040)G_L^{-0.5}V_t^{-0.25} \quad (2.5)$$

where: $\lambda_1$ is in cm,

$G_L$ is in °C/cm, and

$V_t$ is in cm/s.

Equation 2.4 can be approximated by:

$$\lambda_1 = 0.0722G_L^{-0.5}V_t^{-0.25} \quad (2.6)$$

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The linear dependence of the PDAS on $G_L^{-0.5}V_i^{-0.25}$ as predicted by Hunt's model is still valid for the complex alloy PWA1480 but the magnitudes are about 15% lower. Trivedi's analytical model shows excellent agreement with the experimental results as illustrated in Figure 2-26. These results show that predictions of PDAS in complex superalloys using analytical models developed for simple binary alloys are valid.

2.3 DS Furnaces

This section describes typical DS furnace arrangements, along with how they establish the thermal gradient across the solidifying interface and how it is translated along the length of the casting. As discussed in Section 2.2, the magnitude ($G_L$) and rate of travel ($V_i$) of the gradient controls the resulting microstructure of the solidified material. Therefore, a DS furnace arrangement should provide for an adequate measure of control over $G_L$ and $V_i$ to ensure that the desired microstructure forms during casting. This section follows the approach used by McLean [4]: the types of DS arrangements will be divided into those with both the furnace and the mold stationary and those with a moving furnace or mold. The description also roughly follows the chronological development of DS furnaces, as they were used to produce turbine blade components. In general, these developments were aimed at:

1. Achieving a constant $G_L$ – to maintain a constant microstructure throughout the component.

2. Increasing $G_L$ so that a higher $V_i$ was possible – to produce a finer microstructure with improved mechanical properties.
3. Achieving independent control of $G_L$ and $V_1$ — to maintain the required microstructure throughout the casting and to provide for economical operation (e.g. higher $V_1$ to reduce casting times).

2.3.1 Stationary Furnace and Casting

The simplest DS “furnace” consists of a mold with one chilled end and thermal insulation elsewhere, Figure 2-27 [4]. Solidification starts when a molten charge is poured into the mold and is cooled by the chill. Solidification is completely predetermined by the size and geometry of the component, the physical and thermal properties of the alloy, mold, and chill, and the initial temperature of the melt and mold. Once cast no control over the temperature gradient or rate of solidification is possible. During solidification the liquid-solid interface moves away from the chill and, consequently, slows down. The thermal gradient is also reduced. Therefore, the microstructure varies throughout the casting and cannot be used in practical applications.

A slight variation of the above involves casting a superheated melt into a heated mold and then gradually reducing the mold temperature in a controlled manner. Since the solidification velocity is dependent on the difference between heat input to the mold and heat output from the mold, it can be maintained at a constant value by controlling the heat input. There are several ways of doing so, and two examples are to use exothermic materials to surround the mold and the power down method. The use of exothermic materials to surround the molds is shown schematically in Figure 2-28 [31]. The “Exomold” exothermic material is used to provide a temperature gradient — hence it is
thicker near the top of the casting. The water-cooled copper chill plate was used to provide for directional heat removal. This set up provided for some measure of control over the cooling rate, and thus the solidification velocity. However, low temperature gradients and the inability to control them after casting the melt led to too much microstructural variability for the process to be used for precision castings or large components. In the power down process, varying the power to a furnace external to the mold controlled heat input. The casting temperature could be monitored and the rate of power reduction could be modified as necessary to give a steady solidification rate. Resistance heaters were initially used to heat the mold, Figure 2-29 [35]. Induction heating simplified the process by allowing the use of conventional investment casting molds, Figure 2-30 [35]. Two separate zones were used to heat the mold to the required temperature for casting. Once the melt was cast into the mold, the bottom zone was turned off, creating a temperature gradient along the length of the component. Heat was extracted from the chill end of the component, and the solidification front moved away from the chill. As in the simple case, this led to a reduction in the gradient, resulting in slower rates as solidification proceeded. Attempting to increase the solidification rate by reducing the power input faster led to a further drop in the gradient. For a given gradient, once the solidification rate increased above a critical value (Figure 2-23), a transition from columnar to equiaxed growth occurred, limiting the length of a component that could be directionally solidified. If higher temperature gradients could be attained, higher solidification rates could be used, and aligned columnar structures could be maintained over much longer components. This was made possible by the Bridgman crystal growing technique, and is described in Section 2.3.2.
2.3.2 Moving Furnace or Casting

There are three general categories of DS furnaces where either the furnace or casting moves relative to each other [50]:

1. Normal freezing (Bridgman method) – the entire charge is melted and then solidified from one end.

2. Czochralski method – a large charge is melted and a small crystal is withdrawn from it.

3. Zone melting – only a small zone of the charge is molten at any one time.

In the Bridgman method, a charge is melted in a long, small cross-section mold and then withdrawn from the furnace until the charge is solidified. A similar method uses a horizontal boat. The Czochralski method, also known as crystal pulling, is widely used to grow single crystals of silicon, germanium, and non-metals. A seed crystal, attached to a vertical pull rod, is lowered into a large, molten charge, allowed to equilibrate, and then raised slowly. Solidification proceeds from the seed crystal as it is withdrawn. In the zone melting method, the whole charge is not molten at one time. A small portion of the charge is melted, typically with induction heating coils. This molten “zone” is passed through the charge by moving the induction coil or the crystal itself. If no crucible is used, as is widely done when melting reactive or high melting temperature materials, this process is known as the floating zone method. This process is very useful on a laboratory scale, but is limited by the small charge size and the fact that component shapes cannot be produced.
Most current laboratory and commercial DS processes for superalloys are variants of the Bridgman crystal growing technique [4]. As mentioned above, the furnace and the casting are moved relative to each other – effectively removing the casting from the hot zone of the furnace. Compared to the power down process, the cooling of the casting is more efficient due to the radiation heat transfer from the casting to the wall of the cold zone of the furnace. The conduction of heat to the chill dominates at the start, but radiation heat transfer becomes more important as solidification proceeds. A steady state is reached when the dominant mechanism for heat loss is radiation. To maximize the temperature gradient, radiation baffles separating the hot and cold zones of the furnace are used to minimize radiant heat losses from the hot zone to the cold zone.

A Bridgman furnace is illustrated in Figure 2-31 [38] and the process is shown schematically in Figure 2-32. The liquid-solid interface is located at a distance \( x \) above the junction of the hot and cold zones. The withdrawal speed of the casting is \( V_w \). At steady state, the solidification velocity, \( V_h \), is assumed to be the same as \( V_w \). As \( V_l \) is increased the liquid-solid interface moves closer to the junction of the two zones, \( x \) decreases). For DS growth to occur there must be longitudinal heat flow, due to conduction along the solid ingot, at the liquid-solid interface. This restricts the liquid-solid interface to the hot zone \( (x>0) \). Otherwise there would be radial heat transfer in the casting, causing non-directional growth such as equiaxed grains nucleating and growing along the walls of the mold. This limits the maximum possible solidification velocity,
$V_{\text{max}}$, for directional growth when $x=0$ (c.f. Section 2.2.1). For the steady state, McLean [4] has shown the maximum velocity for a cylindrical ingot to be:

$$V_{\text{f max}} = \left[ \frac{2\alpha \sigma \varepsilon}{r \rho f L_f} \left( T_m^4 - T_o^4 \right) \right]^{1/2}$$

(2.7)

where: $\alpha$ = absorptivity of the surroundings

$\sigma$ = Stefan-Boltzmann coefficient

$\varepsilon$ = emissivity of the ingot

$r$ = radius of the ingot

$T_m$ = alloy melting temperature

$T_o$ = heat sink temperature (cold zone walls)

Using Equation 2.1 and setting $G_L=0$, Flemings [50] calculated the maximum velocity, $V_{\text{l max}}$, to be:

$$V_{\text{l max}} = \frac{K_s G_s}{\rho_s L_f}$$

(2.8)

McLean [4] also calculated the temperature gradients in the solid and liquid parts of the ingot, $G_s$ and $G_L$ as:

$$G_s = \frac{2\alpha \sigma \varepsilon}{V_f K_s h} \left( T_m^4 - T_o^4 \right)$$

(2.9)

and

$$G_L = \frac{1}{K_L} \left[ G_s - \rho L_f V_f \right]$$

(2.10)
These equations reveal a good deal about this DS process. Firstly, $G_L$ is sensitive to both $V_1$ and $r$. Slower $V_1$ (c.f. Section 2.2.1) and small ingots result in higher gradients. Once the maximum solidification rate is exceeded, $G_L$ becomes negative, leading to the breakdown of directional growth, as described above. The maximum possible steady state temperature gradient is determined by the radiation characteristics in Equation 2.9.

To improve temperature gradients further could only be accomplished by replacing the radiation cooling with a more effective heat transfer mode such as conduction. This has been achieved by methods such as liquid metal cooling [63], where the mold is withdrawn into a pool of a low melting point metal such as tin. This method has several advantages (interdependence of temperature gradient and solidification rate and higher possible temperature gradients allowing faster growth rates), but is quite difficult to use in a production capacity.

Bridgman DS furnaces are, in some respects, similar to conventional equiaxed investment casting furnaces, but incorporate such features as zone heaters, motors for mold withdrawal, and water-cooled copper chills [38]. Zone heaters ensure large temperature gradients are achieved. Resistance heating elements or graphite induction heaters are generally used, and typically have two or more independently controlled zones. The motors that are used to withdraw the molds must be extremely low vibration units, especially for SX casting. The copper chills promote the start of directional solidification. In the past, DS products were processed in modified equiaxed casting furnaces; newer furnaces are designed and built exclusively for DS processing.
The current method of DS/SX processing is, generally, as follows:

1. A thin-wall investment casting mold, open at the top and bottom, is placed on a water-cooled copper chill.
2. The mold is placed into an induction heated vacuum furnace. It is heated by radiation above the liquidus temperature of the alloy to be cast.
3. The molten charge is cast into the mold and allowed to thermally stabilize.
4. Some growth starts at the chill, which is used to ensure good nucleation of grains.
5. The grains grow and crowd each other out until only grains with the preferred growth direction remain to grow through the casting. In SX casting, a grain selector or a seed crystal is used to only allow one grain to grow into the component cavity.
6. The mold is withdrawn from the heated zone of the furnace into the cold zone of the furnace to enhance heat transfer and the thermal gradient. The withdrawal is done at a rate so as to maintain columnar growth.

2.4 DS Processing of γ-TiAl Alloys

As mentioned in Section 1.3, γ-TiAl alloys are candidate materials to replace superalloys in some gas turbine engine component applications such as LPT blades. The application of these alloys to date has been prevented by a poor balance of mechanical properties. Microstructural control through DS processing is thought to be one of the most promising methods of achieving an adequate balance of mechanical properties. This section begins
with a brief review of γ-TiAl alloy fundamentals, focusing on the fully lamellar microstructure. It will concentrate on the fully lamellar microstructure found in PST crystals, rather than in polycrystalline material, as it relates more directly to DS processing. The discussion will then move to the current status of DS processing of these alloys – in particular the methods used to align the lamellar microstructure.

2.4.1 γ-TiAl Alloy Fundamentals

γ-TiAl alloys of engineering importance are two phase alloys that contain approximately 45-48 at. % Al and 0.1-8 at. % (all subsequent percentages will be given in at. %, unless otherwise noted) of secondary alloying elements such as Cr, V, Mn, Nb, Ta, W, Mo, Si, C, and B. The two phases that these alloys consist of are the ordered face centered tetragonal gamma (γ) phase –TiAl and the ordered hexagonal alpha-2 phase (α₂) – Ti₃Al. Figure 2-33 shows the region of interest for γ alloys in the binary Ti-Al equilibrium phase diagram [64].

The microstructures, and hence the mechanical properties of these alloys, are very sensitive to the alloy composition and processing history. The typical microstructures formed are illustrated in Figure 2-34 [6] and can be classified into four groups: fully lamellar (FL), nearly lamellar (NL), duplex (DP), and near-gamma (NG). FL microstructures consist of alternate layers of γ and α₂ plates of different crystal orientations. NL structures are similar to FL ones, except for finely dispersed γ grains within the FL matrix. The DP structure is the finest, containing a fine mix of equiaxed γ
and lamellar grains. The NG structure is mostly equiaxed γ with a fine dispersion of α₂ structures. Generally, the DP structure has better tensile properties while the FL structure has better fracture toughness and creep properties. FL structures have attracted the most attention recently because of their better fracture toughness and high-temperature properties.

2.4.1.1 Polysynthetically Twinned Crystals

Gamma alloy ingots produced by ingot metallurgy techniques generally have a polycrystalline FL microstructure [65], Figure 2-34(a). If these ingots are remelted and directionally solidified at the correct rate, a single-crystal-like set of specifically oriented lamellae is formed [19]. These have been termed polysynthetically twinned (PST) crystals, after the phenomena of polysynthetic twinning found in mineral systems. PST crystals have been used to study the deformation behaviour of the major-constituent γ phase and the lamellar structure itself. PST crystals are typically grown using an optical floating zone furnace [20]. The FL microstructure of a PST crystal is shown in Figure 2-35 [19]. The FL structure of PST crystals is described in detail below.

2.4.1.2 Formation and Characteristics of the Fully Lamellar Microstructure

The formation of the FL structure can be explained with reference to Figure 2-33. In the composition range of interest, 45-48% Al, solidification proceeds via two peritectic reactions, L+β→α and L+α→γ, or the single peritectic, L+α→γ. The composition and the cooling rate determine the exact route that is followed. The FL structure forms during cooling within the α+γ phase field. γ precipitates from α producing a lamellar structure
of transformed $\gamma$ and the remaining $\alpha$. Upon cooling below the eutectoid, $\alpha \rightarrow \alpha_2 + \gamma$, additional $\gamma$ is formed by the thickening of the existing $\gamma$ or by the creation of new lamellae. The ordering of $\alpha$ to $\alpha_2$ generally occurs after the formation of the lamellar structure. The final lamellar structure consists mostly of $\gamma$ lamellae, with about 3-15 volume % of $\alpha_2$ lamellae. $\gamma$ lamellae are typically 0.5-2 $\mu$m thick. $\alpha_2$ lamellae are typically 0.5 $\mu$m thick, are separated from each other by $\gamma$ lamellae, and are usually 10 $\mu$m apart. Note that these lamellar dimensions are very dependent upon processing and composition.

The formation of $\gamma$ is such that the close-packed planes and directions are parallel to the corresponding planes and directions in the $\alpha$ phase, $(111)_\gamma // (0001)_{\alpha_2}$ and $<110>\gamma // <1120>_{\alpha_2}$ [20]. The mixed notation $<110>$ is used to differentiate the first two indices from the third index which does not play the same role as the first two. While the three $<1120>$ directions in the hexagonal structure are equivalent, the $[110]$ direction in the face-centered tetragonal structure is not equivalent with the $[011]$ and $[101]$ directions due to the slight tetragonality of the $\gamma$ structure. This results in six possible orientation variants, or, ordered domains, corresponding to the six possible orientations of $<110>$ on $(111)$ in the $\gamma$ phase with respect to $<1120>$ on $(0001)$ in the $\alpha_2$ phase. Therefore, in addition to the $\gamma/\alpha_2$ lamellar boundaries, the $\gamma$ lamellae between the $\alpha_2$ lamellae form an ordered domain structure with various orientation relationships between neighbouring domains. Each of the $\gamma$ lamellae corresponds to one of the six possible orientation variants, and three different types of intervariant boundaries may be formed (depending
on the stacking sequence of the different orientations): $\gamma'/\gamma$ true-twin domain boundaries, $\gamma'/\gamma$ pseudo-twin domain boundaries, and $\gamma'/\gamma$ 120° rotational order-fault domain boundaries. Ordered domains with different orientations also coexist within each of the $\gamma$ lamellae. The lamellar domain boundaries are flat and parallel but domain boundaries within each of the $\gamma$ lamellae do not have any preference for certain crystallographic planes.

2.4.1.3 Mechanical Properties of the Fully Lamellar Microstructure

The mechanical properties of PST crystals are very anisotropic with respect to the lamellar orientation. They show a strong dependence on $\phi$, the angle between the lamellar boundaries and the loading axis, as illustrated in Figure 2-36 and Figure 2-37 [19] for yield stress and ductility. The following briefly reviews the mechanical properties of the FL microstructure, and will highlight the dependence of tensile (yield stress and ductility), fatigue and fracture, and creep properties on the lamellar microstructure.

The yield stress of PST crystals is high when the lamellae are parallel or normal to the loading axis ($\phi = 0^\circ$ or 90°) and is low when the lamellae are in intermediate orientations ($30^\circ < \phi < 60^\circ$). Because of this behaviour, the 0° or 90° orientation is known as the hard mode of deformation and the intermediate orientations are termed the easy mode of deformation. As might be expected, the ductility is low in the hard mode of deformation and high in the easy mode. In the normal orientation ductility is practically 0 % but can, however, be quite high in the parallel orientation with values as high as 10 % having been
reported. This behaviour can be explained by examining the strengthening effects of the
different boundaries that exist in the fully lamellar microstructure of two-phase gamma
alloy PST crystals [66,67].

As mentioned in Section 2.4.1.2 there are four types of boundaries in the lamellar
structure and these boundaries cause resistance to deformation. In the hard mode
deformation occurs across the lamellar boundaries and all four boundaries can resist the
deformation. In the easy mode deformation occurs mostly in the γ phase parallel to the
lamellar boundaries and only the γ/γ 120° rotational order-fault domain boundaries resist
deformation. The yield stress in the hard mode is consequently higher than in the easy
mode because more strengthening mechanisms are active. In addition, the γ/α2 phase
boundaries provide the most resistance to deformation while the γ/γ 120° rotational order-
fault domain boundaries provide the least as they only change the direction of shear
deformation. It is also interesting to note from Figure 2-36 that the yield stress in the
normal orientation is significantly higher than in the parallel orientation. This has been
attributed to the occurrence of different modes of deformation in the α2 phase – prismatic
versus pyramidal slip for the parallel and perpendicular orientations, respectively [68].
The yield stress dependence on lamellar spacing is similar to the Hall-Petch relationship
[68]. Yield stress increases with decreasing lamellar spacing, Figure 2-38, due to the
presence of more lamellar boundaries. These boundaries are similar to grain boundaries
in hindering the motion of dislocations so a refinement in their spacing will result in
increasing strength. A similar effect occurs for decreasing domain size within individual
γ lamellae.
Binary PST crystals are less ductile when tested in air or hydrogen than when tested in vacuum or dry air. The fracture surfaces of specimens tested in air or hydrogen are cleavage-like cracks that lie parallel to the lamellar boundaries. The fracture surfaces of specimens tested in vacuum or dry air occur in a zigzag manner across the lamellar boundaries. The change in fracture mode is believed to be due to hydrogen embrittlement. ψ/γ pseudo-twin and 120° rotational order-fault domain boundaries are susceptible to hydrogen attack, resulting in lower bond strength along these boundaries that leads to the cleavage-like failures.

The fracture toughness, fatigue crack growth, and creep properties of the FL structure are also highly dependent on the morphology and the orientation of the lamellar boundaries to the loading axis [69,70,71,72,73]. Fracture toughness depends on the size of the lamellar grains, the lamellar spacing, and the angle between the crack growth plane and the lamellae [69,70]. Increasing grain size results in increasing toughness, but also leads to a decrease in ductility. However, ductility and toughness may be balanced with a lamellar structure of refined lamellar grains, maintaining reasonably high levels of both toughness and ductility. A finer lamellar spacing increases toughness because α3/γ lamellar and γ/γ domain boundaries act as effective barriers to crack propagation. In general, there are three possible orientations of a crack with respect to the lamellae, as illustrated in Figure 2-39. The first type, Figure 2-39 (a), is the crack delamination orientation. Test specimens with this orientation fail in a cleavage-like mode along planes parallel to the lamellae and exhibit low fracture toughness. The other types,
Figure 2-39 (b) and Figure 2-39 (c), are the crack divider and crack arrester, respectively. Fracture surfaces of test specimens with these orientations are much rougher. Their fracture toughness is much higher due to toughening mechanisms such as crack tip branching. As a crack propagates delamination of the lamellae occurs ahead of the crack tip. For the crack to continue propagating a new crack tip must be formed. The fatigue strength is also dependent on the lamellar orientation. Test specimens with lamellae parallel to the loading direction have higher fatigue strength than specimens with lamellae at 45° angles to the loading direction [71]. Creep properties have also been shown to depend on the lamellar morphology. Generally, a FL lamellar structure with fine grain size and lamellar spacing has the best resistance to creep deformation [72,73].

2.4.1.4 Effects of Composition and Processing on the Fully Lamellar Microstructure

The microstructure and resultant mechanical properties of γ-TiAl alloys are heavily dependent upon the alloy composition and processing parameters. Some of the effects of composition and processing on the FL microstructure are discussed here, including Al content, ternary alloying additions, and DS processing parameters.

The Al content of a γ-TiAl alloy is very important; small changes can drastically effect tensile properties [9,68]. This is illustrated for a polycrystalline investment cast alloy in Figure 2-40 and for a PST crystal in Figure 2-41. The yield stress behaviour may be attributed to the decrease in lamellar spacing that is associated with decreasing Al content. A fine lamellar spacing would then be desirable to increase strength,
necessitating a low Al content. This would then, as illustrated in Figure 2-40, lower the
ductility. However, it has been reported that good ductility can be achieved with an
oriented, uniform, and fine lamellar structure. This is likely due to the absence, in a
correctly oriented microstructure, of the normal lamellar orientation that is closely related
to the brittleness of the polycrystalline lamellar structure [65].

Ternary alloying additions can have significant effects on the mechanical properties, such
as tensile strength, of PST crystals as illustrated in Figure 2-42 [65]. Three important
facts should be noted from this figure. Most importantly, the yield stress of ternary PST
crystals is always higher than that of binary PST crystals in all lamellar orientations. The
most effective strengtheners overall seem to be Mo, Ta, and Nb. Because more
information was available in the literature regarding the specific effects of Mo and Nb,
further discussion will focus on these alloys. The increase in strength results from solid
solution strengthening of the $\alpha_2$ phase [6] and a decrease in the domain size within $\gamma$
lamellae [74], increasing the number of $\gamma/\gamma$ 120° rotational order-fault boundaries, Figure
2-43. Secondly, the strengthening effect is not the same for different lamellar
orientations. The hard orientations (lamellae at 0° or 90° to the loading direction) have a
larger increase in strength than the soft orientations as a result of having more boundaries
to resist deformation. In addition, the boundaries active in the hard mode are more
effective than the boundaries in the soft mode, as discussed in Section 2.4.1.3. Lastly,
some ternary additions are more effective in strengthening the hard orientation than the
soft orientation, and vice versa. For example Mo is a more effective strengthener than
Nb in the hard mode of deformation while Nb is more effective in the easy mode. This
might occur because Mo enriches the $\alpha_2$ phase. Since the $\alpha_2$ lamellae are the most
effective strengtheners for the hard mode, then Mo is a better strengthener for the hard
mode. Conversely, Nb enriches the $\gamma$ phase and causes an increase in the number of $\gamma/\gamma$
120° rotational order-fault boundaries. This behaviour would be useful if these
strengthening effects are independent. A quaternary alloy with both Mo and Nb additions
would then have improved yield stress over a range of lamellar orientations. There was
nothing found in the current literature to support or refute this behaviour.

Ternary additions of Mo and Nb have also been shown to increase the ductility of PST
crystals [65,74,75]. In the case of Mo, at least, this behaviour is thought to be due to a
reduction of environmental embrittling effects [75] that were mentioned in Section
2.4.1.3. The domain boundaries that are susceptible to hydrogen attack may also accept
ternary alloying atoms. It is thought that Mo atoms preferentially segregate to these
boundaries, reducing their susceptibility to hydrogen attack. PST crystals with additions
of Mo tested in air have fracture surfaces similar to binary PST crystals tested in vacuum
or dry air, Figure 2-44. The increase in ductility of the crystals with ternary additions is
then seen to be due to a change in fracture mode.

The growth rate has an important effect on the lamellar structure and the resulting
mechanical properties also. In superalloys, dendrite morphology is known to become
finer as heat is extracted at a greater rate during solidification [4,50]. In the case of $\gamma$-
TiAl alloys, the lamellar structure becomes finer as heat is extracted at a greater rate
[16,68,76]. The results of Takeyama et al. [16] show the finer lamellar structure to
improve the mechanical properties of γ-TiAl alloys, Figure 2-45. Faster growth rates resulted in finer lamellar spacing and finer α₂ lamellae, which led to higher levels of strength and ductility. In particular, region B in Figure 2-45 is attractive due to its higher strength and relatively high ductility. The microstructure in region B was composed of relatively fine columnar grains, while the microstructure in region A was single crystal-like, demonstrating a fine-grained columnar structure with FL microstructure has the capability to achieve a balance of mechanical properties.

2.4.2 Why DS Processing?

DS processing could be a powerful method of controlling the lamellar structure of γ-TiAl alloys, thus achieving an adequate balance of mechanical properties and allowing the service stresses and temperatures of γ-TiAl to be increased. As mentioned in Section 1.3, it was the research on PST crystals, aimed at gaining a better fundamental knowledge and understanding of the microstructural characteristics and mechanical properties of the fully lamellar microstructure, that led to interest in the DS processing of γ-TiAl alloys. From the review of the FL microstructure above it is apparent that to achieve a good balance of mechanical properties a relatively fine-grained structure is required with fine lamellar spacing and lamellae oriented parallel to the main stress axis. Such a structure would have a good combination of strength, ductility, toughness, fatigue, and creep properties. A target microstructure with these properties has been proposed by Yamaguchi et al. [19,77], and is described as:

- Columnar grains with FL microstructure.
- γ/α₂ and γ/γ lamellar boundaries parallel to the main stress axis.
- Individual grains rotated slightly about their longitudinal axes to provide for a level of off-axis fracture toughness.

A schematic of this microstructure is illustrated in Figure 2-46. The results of some promising experimental work, reviewed in the following section, have demonstrated that it may be possible to achieve this structure through DS processing.

2.4.3 The Current Status of DS Processing of γ-TiAl

This section reviews the current status of research on DS processing of γ-TiAl that is based on the previous work on PST crystals. However, before the knowledge gained from studying PST crystals can be used to develop commercially manufactured DS γ-TiAl components some fundamental problems must be overcome.

One difficulty lies in finding a suitable mold/facecoat material that is sufficiently stable in contact with molten γ-TiAl alloys. In DS processing the mold and melt will be in contact for relatively long periods of time, and excessive reaction between them must be minimized. In the conventional casting of γ-TiAl alloys, oxygen pick-up indicates excessive reaction between the mold and the melt has occurred; levels in excess of 1000 ppm, by weight, are unacceptable [14]. Several different mold systems have been used to DS process γ-TiAl alloys, as reported in the literature. London et al. used a specifically designed ceramic mold system that was based on a traditional investment casting mold material used for processing Ti-based alloys [14]. The O₂ content of DS processed γ-TiAl was found to range from 1200 to 1800 ppm. While higher than desirable, the levels
were much lower than those found in castings processed in the traditional investment casting molds. Huang has shown that a CaO crucible can be used to melt γ-TiAl alloys with acceptable levels if the composition of the alloy is tailored to reduce O₂ pick-up [78]. Johnson et al. have used both CaO and Y₂O₃ molds [79]. In the case of CaO, no severe mold-melt reaction was observed. However, CaO is sensitive to moisture pick-up and is difficult to handle in production environments [14]. The extent of the reaction with the Y₂O₃ mold and the melt was not discussed. As an initial part of the current overall project a Y₂O₃ mold system was chosen as the most stable in contact with molten γ-TiAl alloy [80]. This mold system has been used to produce several DS γ-TiAl alloy castings with no severe mold-melt reactions. Currently, molds made from Y₂O₃ seem to be adequate but have only been manufactured in simple cylindrical geometry. Whether or not this material can be used to form complex investment casting molds with the same level on non-reactivity with the melt remains to be determined.

Perhaps the most difficult problem to be overcome is the tendency of γ-TiAl alloys to form the disadvantageous lamellar orientation after solidification and cooling. For many cast γ-TiAl alloys the lamellar structure forms perpendicularly to the growth direction, resulting in an extremely brittle material. A favourably oriented microstructure is difficult to obtain because the lamellar structure forms from solid state reactions and not directly from the liquid [77]. Two methods of aligning the lamellae with the growth direction during DS have been shown, at least in some degree, to work in obtaining a favourably oriented microstructure. The methods are: (1) choosing an alloy composition whose microstructure transforms to a correctly oriented one upon solidification and
subsequent cooling or (2) using a seed material to force the correct microstructure to form upon solidification and cooling. These methods are reviewed in the following two sections.

2.4.3.1 Microstructure Control Through Choice of Alloy Composition

One method of aligning the lamellae with the growth direction during DS is to choose an alloy with a composition that has the correct solidification pathway that results in the final structure having the desired orientation [75,81,82,83]. However, this is difficult as the lamellar structure forms from solid state reactions and not directly from the liquid [77]. Examining the solidification pathways and solid state reactions of two alloys with compositions C1 and C2, Figure 2-47, illustrates this difficulty. For alloy C1 the $\beta$ (body centered cubic, BCC) phase is the primary solidification phase and its preferred growth direction is $<100>$ [37]. As cooling proceeds, the $\alpha$ phase nucleates on the close-packed planes of the $\beta$ phase with the relationship $(0001)_\alpha \parallel (110)_\beta$. Upon further cooling, $\gamma$ nucleates from the $\alpha$ with the relationship $(111)_\gamma \parallel (0001)_\alpha$ and the $\alpha$ phase subsequently orders to the $\alpha_2$ phase. Because of these relationships the $\gamma/\alpha_2$ and $\gamma/\gamma$ lamellar boundaries will be parallel or at 45° angles to the growth direction. Under equilibrium solidification conditions this would result in 1/3 of the boundaries parallel to the growth direction and 2/3 of the boundaries at 45° angles to the growth direction. For alloy C2 the $\alpha$ phase is the primary solidification phase and its preferred growth direction is [0001]. As cooling proceeds, the $\gamma$ phase nucleates on the close-packed planes of the $\alpha$ phase with the relationship $(111)_\gamma \parallel (0001)_\alpha$ and the $\alpha$ phase subsequently orders to the $\alpha_2$ phase. Because of these relationships the $\gamma/\alpha_2$ and $\gamma/\gamma$ lamellar boundaries will be
perpendicular to the growth direction. Therefore, depending on the composition of the alloy, the resulting lamellar microstructure would have a mix of parallel and 45° boundaries or perpendicular boundaries.

The most important factor in controlling the lamellar orientation through compositional control is that the liquid must fully transform to the β phase during solidification [81]. If any part of the melt solidifies directly as the α phase it will have lamellae oriented normally to the growth direction. To ensure that the β phase is the primary solidification phase detailed information is needed regarding the phase transformations that occur in γ-TiAl alloys, especially near their melting temperatures. This, however, is a problem as even the binary Ti-Al phase diagram at high temperatures has not been precisely defined, let alone ternary Ti-Al-X phase diagrams. Kim *et al.* [81] confirmed the binary phase diagram proposed by Okamoto [84], Figure 2-48. For binary γ-TiAl alloys to fully transform to the β phase from the liquid the Al content must be below 45%. Higher Al contents will have some primary α phase formation, and consequently, some unfavourably oriented lamellae. Kim *et al.* verified this behaviour, as reviewed below.

Kim *et al.* [81] directionally solidified several different γ-TiAl compositions at different withdrawal rates. Two ingots with compositions of Ti-44Al and Ti-48Al were DS processed at a withdrawal rate of 90 mm/h. The resulting microstructures are shown in Figure 2-49. The lamellae were aligned in various orientations ranging from 0° to 45° to the growth direction in the Ti-44Al alloy. They were normal to the growth direction in the Ti-48Al alloy. This verified that an Al content below 45% results in a favourably
oriented lamellar structure. However, an Al content below 46% is known to adversely affect ductility – Shimada [85] has shown that a favourably oriented structure in a Ti-45Al alloy had only 1% tensile elongation. Kim et al. DS processed several ternary alloys of composition Ti-46Al-2X, with X being a β phase stabilizer: Mo, Cr, or Nb. The stabilizers were used to extend the β phase field to higher Al contents. The Al content was fixed in the range of 46-48% that has been reported to yield good ductility in ternary alloys [86]. The resulting microstructures for a withdrawal rate of 90 mm/h are illustrated in Figure 2-50. For all compositions the lamellae were oriented at angles ranging from 0° to 45° to the growth direction. This demonstrated that the use of β phase stabilizers extend the β phase field to higher Al contents, allowing the liquid to fully transform to the β phase. It also demonstrated that a reasonably well-aligned lamellar structure could be formed by choosing a correct alloy composition.

During these experiments it was found that the withdrawal rate had an affect on the lamellar orientation. A Ti-47.5Al-2.5Mo alloy solidified at 90 mm/h had a favourably oriented lamellar structure. However, when the withdrawal rate was increased to 180 and 360 mm/h, the lamellae were normal to the growth direction. This behaviour is illustrated in Figure 2-51. It was proposed that this was caused by a shift of the L + β → β transus line to a lower Al content with increase in growth rate, and is illustrated on the binary Ti-Al phase diagram in Figure 2-52. This would cause the formation of primary α phase, which would then transform into the α2 phase and have the unfavourable normal lamellar orientation.
Johnson et al. also attempted to achieve a favourably oriented microstructure through compositional choice [82,83]. A Ti-45Al alloy was DS processed over a range of withdrawal rates from 40 to 200 mm/h [82]. The resulting lamellar structure had a range of orientations with lamellae at angles from 0° to 45° to the growth direction. A Ti-46Al alloy was also solidified at a withdrawal rate of 200 mm/h [82]. The resulting microstructure consisted of several large lamellar grains with lamellae oriented parallel to the growth direction. The tensile elongation of these structures was found to be quite low, and was attributed to the large grain size and high volume fraction of the α₂ phase. Johnson et al. then attempted to overcome this by DS processing TiAl-Mo-B alloys [83]. Mo was used to stabilize the Β phase and provide for a level of solid solution strengthening. B was used to refine the solidification microstructure. For some compositions and processing conditions, a well-aligned structure resembling the target microstructure was produced and had good initial mechanical properties: compressive yield strength greater than 500 MPa at 800°C and 2% room temperature ductility.

2.4.3.2 Microstructure Control Through Use of a Seed Material

The other method that has been used successfully in controlling the lamellar microstructure of γ-TiAl alloys is through the use of a seed material. Johnson et al. have performed a large volume of research on this method, as briefly reviewed below [19,77,79,82,87,88,89]. This research originated from studies on the effects on Si additions to γ-TiAl alloys [77]. Their goals were to determine the solidification sequence of these alloys and to apply that knowledge to produce DS ingots with the target microstructure of Figure 2-46. In the course of the work it was noticed that some of the
DS ingots had only one lamellar orientation, perpendicular to the growth direction in all grains. This led to the hypothesis that microstructural control might be obtained by using a seed crystal with lamellae oriented parallel to the growth direction.

Two requirements must be met in order to control the lamellar orientation with a seed crystal during DS processing [77]:

- The primary solidification phase must be $\alpha$.
- The $\alpha$ and $\alpha_2$ phases must be stable on heating and cooling.

The first ensures the correct lamellar orientation occurs – the high temperature $\alpha$ phase determines the final lamellar orientation, as discussed in Section 2.4.1.2. The second insures the lamellar orientations of the seed crystal and the DS ingot are not lost upon heating and cooling. During heating into the $\alpha + \gamma$ two-phase region, the lamellar structure of the seed must remain stable – $\alpha_2$ simply disorders to $\alpha$. Upon further heating the volume fraction of $\alpha$ increases by the thickening of the $\alpha$ lamellae, not by the nucleation of new $\alpha$ grains. Once in the $\alpha$ single-phase region, the orientation of $\alpha$ lamellae is the same as the original $\alpha_2$ orientation. The second requirement basically means that the seed material cannot pass through the $\gamma$ single-phase region upon heating and cooling. Recrystallization may otherwise occur, resulting in the loss of the desired lamellar orientation. These conditions cannot be met in the binary Ti-Al system due to the relative positions of the $\alpha$ and $\gamma$ phase fields [87]. However, they were met in a small region of the Ti-Al-Si ternary system, illustrated in Figure 2-53, by an alloy with a composition of Ti-43Al-3Si [77].
Johnson et al. performed a series of solidification trials to verify the above hypothesis [77]. A section of a previously DS processed ingot with the chosen composition was cut and rotated 90° to align the lamellae in the desired orientation. The experiment was successful in aligning the lamellae parallel to the growth direction for the complete ingot, Figure 2-54. However, the target microstructure was not achieved as all the grains had the same lamellar orientation. A further trial with a composite seed composed of two crystals joined together was performed. This was also successful in correctly orienting the lamellae as illustrated in Figure 2-55. The ductility of these ingots was poor because the relatively high levels of Si resulted in the formation of very large and brittle silicide (Ti$_2$Si$_3$) particles. Another series of solidification trials were performed to see if microstructural control could be achieved in alloys with compositions differing from that of the seed [79,88,89]. These trials were, again, successful. It was shown that the lamellar microstructure of γ-TiAl alloys with compositions different than the seed material could be controlled. Alloys that would normally solidify in the primary β phase could also be successfully aligned. In these cases, the composition of the initial liquid close to the seed is rich enough in Al and Si that the nucleation of the primary β phase is prevented, allowing the growth of the α phase from the seed material. The choice of alloy compositions with lower Si content resulted in drastically improved ductility.

The above solidification trials were all performed using the zone melting technique described in Section 2.3.2. A more economic and practical technique, however, would be the Bridgman method in which a fully molten charge is cast into a ceramic mold. The
use of a seed crystal in this situation could be difficult as the molten portion of the seed can mix with the entire molten charge [88]. If the molten charge were highly mixed, the solute concentration (Al + Si) could become reduced to a level that would allow some primary β phase to solidify. Seeding will only work if the charge is not stirred and mixing only occurs by diffusion and convection. Solidification trials were performed to see if the seeding technique would work during DS processing by the Bridgman method [79]. A Ti-47Al ingot was produced in a laboratory-scale Bridgman furnace using a Ti-43Al-3Si seed. The entire charge was melted in a CaO crucible with a seed crystal in the bottom and was solidified at a withdrawal speed of 5 mm/h. The resultant microstructure consisted of lamellae parallel to the growth direction and continuous with the seed, Figure 2-56. An ingot with a microstructure resembling the target microstructure was produced by drop-casting Ti-43Al-3Si alloy into a Y₂O₃ crucible containing a composite seed made of four differently-oriented seed crystals and withdrawing at a rate of 25 mm/h. The final microstructure consisted of favourably oriented lamellae, but with the individual grains slightly rotated about their longitudinal axes.
2.5 Summary

The preceding sections highlight the role DS processing played in the development of superalloy gas turbine hot section components, and provide strong evidence as to its future importance in developing $\gamma$-TiAl alloys:

1. A better balance of mechanical properties is needed before $\gamma$-TiAl alloys can realistically replace established superalloys for intermediate temperature structural applications in gas turbine engines.

2. A better balance of mechanical properties can be found in an aligned structure of fully lamellar columnar grains with the lamellar boundaries oriented parallel to the major loading axis of the component.

3. DS processing is a powerful method by which such an aligned microstructure could be formed.

4. Two methods of controlling the resultant DS microstructure are available: correct alloy composition and seeding.
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<th>Hf</th>
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Table 2-1: Representative alloys developed during the last 50 years along with their compositions [3,48].

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<th>Nb</th>
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<th>B</th>
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Table 2-2: The roles of various alloying elements in nickel base superalloys [26].
Figure 2-1: The range of different microstructures that have been developed in superalloys [23].

Figure 2-2: Creep rupture life as a function of $\gamma'$ volume fraction [27].
Figure 2-3: Strength (hardness) of a nickel base superalloy as a function of $\gamma'$ particle size. A peak hardness occurs at a $\gamma'$ particle size of approximately 0.1 $\mu$m [24].

Figure 2-4: Different grain boundary carbide morphologies: (a) favourable discrete, globular carbides [28], (b) unfavourable grain boundary film [29], and (c) unfavourable discontinuous/cellular carbides [29].
Figure 2-5: (a) Waspaloy, a wrought superalloy showing the spheroidal morphology of early, low $\gamma'$ volume fraction alloys. (b) Udiment 700, a cast superalloy showing the cuboidal morphology of later, high $\gamma'$ volume fraction alloys [30].

Figure 2-6: Effect of hafnium addition on creep rupture ductility, at 760$^\circ$C, of superalloy B1900 [3].
Figure 2-7: Creep rupture ductility as a function of temperature. The shaded regions cover the range of ductility for different alloys in the DS and conventionally cast forms [34].

Figure 2-8: Creep curves for MAR-M200 superalloy showing the increase in creep life for DS and SX processed over conventionally cast material [33].
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Figure 2-15: Oxidation resistance of DS MAR M200 + Hf and PWA 1480 (alloy 454) as a function of temperature [45].

Figure 2-16: Schematic of the temperature distribution during directional solidification. 1 corresponds to $V_{l_{\text{max}}}$ and 2 and 3 are decreasing $V_{l}$ [4]. Low values of $V_{l}$ result in high values of $G_{L}$. 

65
Figure 2-17: (a) Equilibrium phase diagram and (b) composition profile across the planar liquid-solid interface of a binary alloy solidifying with equilibrium at the interface [50].

Figure 2-18: Solute redistribution in plane front solidification of a binary alloy with no diffusion in the solid, limited diffusion in the liquid, and equilibrium conditions at the liquid-solid interface. (a) Steady state composition profile. (b) Composition profile at the end of solidification. (c) Equilibrium phase diagram [50].
Figure 2-19: Schematic of the variation in liquidus temperature due to the solute boundary layer in the liquid [4].

Figure 2-20: (a) Stable plane front. (b) Unstable plane front due to constitutional supercooling [50].
Figure 2-21: Schematic of changing solidification morphologies with increasing solidification velocity, \( V_I \) at constant \( G_L \). (a) Plane front. (b) Cellular – cylindrical cells parallel to direction of heat transfer. (c) Cellular – cylindrical cells aligned with the preferred crystallographic growth direction. (d) Cellular/dendritic – flanged cells aligned with the preferred crystallographic growth direction. (e) Dendritic – secondary and higher order dendrite arms have formed [4].

Figure 2-22: Schematic of the effect of increasing solidification velocity, \( V_I \), on the liquidus temperature (\( V_I \) increases from a to d) at a constant gradient, \( G_L \) [4].
Figure 2-23: Morphologies of DS superalloys processed at different solidification velocities, \( V_i \), and gradients, \( G_L \). The lines separating the different morphologies are constant \( G_L/V_i \) contours [4].

Figure 2-24: Dendrite arm spacing vs. cooling rate (\( G_LV_i \)) for several superalloys [57].
Figure 2-25: The primary dendrite arm spacing of three superalloys plotted vs. the solidification parameters as $V_i^{-0.25} G_i^{-0.5}$. The closed symbols represent alloys that solidified with well-defined dendritic structure. The open symbols represent alloys that solidified under plane front conditions [4].

Figure 2-26: The primary dendrite arm spacing of PWA1480 (open circles) plotted vs. the solidification parameters as $V_i^{-0.25} G_i^{-0.5}$. The solid line is the linear least-squared fit to the experimental data, Equation 2.3 [56]. The broken line is the prediction from Trivedi's model [62].
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Figure 2-28: Schematic of DS with exothermic material surrounding the mold and the use of a water-cooled copper chill [31].
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Figure 2-30: Induction heated mold used in the power down process [35].
Figure 2-31: Schematic of Bridgman furnace illustrating radiation cooling and mold withdrawal [38].

Figure 2-32: Schematic of the Bridgman process, showing the steady state temperature profile [4].
Figure 2-33: Binary Ti-Al equilibrium phase diagram showing the region of interest (boxed area) for γ-TiAl alloys [64].
Figure 2-34: The four typical γ-TiAl alloy microstructures: (a) fully lamellar, (b) nearly lamellar, (c) duplex, and (d) near-gamma [6].

Figure 2-35: The lamellar microstructure of a PST crystal [19].
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Figure 2-37: Tensile elongation to failure as a function of the angle between lamellar boundaries and loading axis for binary PST crystals [19].
Figure 2-38: The effect of lamellar spacing on the yield stress in compression of binary PST crystals. Open and closed circles represent results from crystals grown at 2.5 and 5 mm/h, respectively [68].

Figure 2-39: The three possible orientations of a crack with respect to the lamellar orientation: (a) crack delamination, (b) crack divider, and (c) crack arrested [69].
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Figure 2-41: The effect of Al content on yield strength in compression of binary PST crystals. Open and closed circles represent results from crystals grown at 2.5 and 5 mm/h, respectively [68].
Figure 2-42: The effects of ternary additions on the yield stress of PST crystals [65].

Figure 2-43: Domain structure of (a) binary and (b) Mo-doped PST crystals. The average domain size has been approximately halved by the addition of Mo [74].
Figure 2-44: Fracture surfaces of (a) binary and (b) Mo-doped PST crystals tested in air [75]. Binary crystals tested in vacuum or dry air have fracture surfaces similar to (b).

Figure 2-45: Effects of growth rate and Al content on the tensile ductility and ultimate strength of DS processed binary γ-TiAl alloys. Region A has a single crystal-like microstructure while region B is composed of columnar grains [16].
Figure 2-46: Schematic of the target microstructure for DS γ-TiAl alloys that should result in a good balance of mechanical properties [77].

Figure 2-47: Lamellar orientation formation during the solidification and solid state transformations for two binary γ-TiAl alloys with compositions C1 and C2. Their primary solidification phases are β and α, respectively [75].
Figure 2-48: Comparison of the binary Ti-Al phase diagram, near the compositional range of engineering interest, as proposed by (a) Okamoto and (b) Kim et al. [81].

Figure 2-49: Microstructures for (a) Ti-44Al and (b) Ti-48Al ingots DS processed at a withdrawal rate of 90 mm/h. The lamellae are oriented at angles ranging from 0° to 45° to the growth direction for (a) and are normal to the growth direction for (b) [81].
Figure 2-50: Microstructures for (a) Ti-46Al-2Mo, (b) Ti-46Al-2Cr, and (c) Ti-46Al-2Nb ingots DS processed at a growth rate of 90 mm/h. The lamellae are oriented at angles ranging from 0° to 45° to the growth direction for all ingots [81].

Figure 2-51: The effect of increasing withdrawal rates on the lamellar structure of Ti-47Al-2.5Mo ingots from (a) 90 to (b) 180 and (c) 360 mm/h. For (b) and (c) the lamellar orientation is the unfavourable normal one [81].
Figure 2-52: Increasing growth rate shifts the $L + \beta \rightarrow \beta$ transus towards lower Al contents [81].

Figure 2-53: Part of the ternary Ti-Al-Si phase diagram showing the composition range that meets the two necessary requirements of a seed material able to control the lamellar orientation of $\gamma$-TiAl alloys [77].
Figure 2-54: Lamellar structure of a Ti-43Al-3Si seed and ingot showing the successful alignment of the lamellar structure. Arrows indicate Ti5Si3 particles [77].

Figure 2-55: (a) Schematic of the composite seed and resulting ingot illustrating the different lamellar orientations in each crystal (seed) and grain (ingot). (b) Transverse section of the ingot. (c) Longitudinal section of the ingot. [77]
Figure 2-56: Longitudinal section of a Ti-47Al ingot DS processed from a Ti-43Al-3Si seed crystal at a withdrawal rate of 5 mm/h in a CaO crucible [79]. The diameter of the ingot is 16 mm.
3 Thesis Objectives and Scope

This thesis is part of a research partnership program established between Carleton University, the Structures, Materials and Propulsion Laboratory of the National Research Council Canada, and Pratt & Whitney Canada with the ultimate goal of developing a pilot-scale industrial process to manufacture DS γ-TiAl airfoil-shaped structures. In the scope of this overall project, several major tasks necessary to the accomplishment of its goal were identified. They form the specific objectives of this thesis and are:

1. Specification of a dedicated DS facility for γ-TiAl. This facility must be representative of a pilot-scale industrial facility that could be modified and scaled-up for component production purposes.

2. Performance of solidification trials on a nickel-base alloy to demonstrate the capabilities of the facility in terms of solidification parameters such as withdrawal rate, temperature, and temperature gradient and the casting atmosphere, i.e. levels of vacuum and inert gas during casting.

3. Determination of initial γ-TiAl alloy compositions that would be suitable for preliminary DS processing investigations.

The next chapter describes the DS facility installed at Carleton University. The following chapters describe solidification trials with the nickel-base alloy, the choice of an initial γ-
TiAl alloy composition, and the results of preliminary DS casting trials with that composition.
4 Carleton University DS Facility

The dedicated DS casting facility had to meet three main requirements. The first was that columnar grains must be made to grow from one end of the casting to the other. The second was that casting parameters, including temperature, temperature gradient, and growth rate, must be controllable and repeatable. This ensures that the component microstructure formed will be the one desired, uniform throughout, and reproducible. The third was that airfoil-shaped components must be produced. A Bridgman-type furnace was the best way to meet all of these requirements (c.f. Section 2.3): (1) it provides for the establishment and translation of the required temperature gradient, (2) temperature gradients and growth rates can be maintained at constant values throughout the growth process and can be, to some degree, independently controlled, and (3) component shapes can be produced by using investment casting molds.

Of several options to design and assemble or procure an appropriate furnace that were considered the most promising was the purchase of an existing Bridgman furnace from Lehigh University in Bethlehem, PA. The ultimate goal of this project – to develop a pilot-scale industrial process – provided the motivation for choosing this route. It was determined that the purchase of an existing, proven facility could be accomplished in a relatively short period of time, allowing for more developmental work on specific alloy
compositions and processing parameters rather than developing the equipment itself. The furnace was previously used to produce DS and SX-like structures in commercial superalloys [90], and with modification could be used to cast γ-TiAl alloys. Furthermore, this facility was a better example of a pilot-scale industrial furnace than other options considered. Scaling up the equipment and making appropriate modifications could make it suitable for casting in a production-like environment. The decision to purchase the facility was made after examining the facility and observing its operation. The furnace was partly disassembled, transported, and reassembled at Carleton University.

4.1 General Description of the DS Furnace

The DS facility as installed at Carleton University is shown in Figure 4-1 and Figure 4-2. A schematic of the furnace chamber is shown in Figure 4-3. A more detailed assembly drawing of the furnace chamber is shown in Figure 4-4. Centorr Vacuum Industries, Nashua, NH, originally manufactured the furnace. The furnace chamber is constructed of stainless steel with a cooling water jacket. The upper chamber is the furnace hot zone. Heating is provided by two independently controlled tungsten mesh heating elements. The upper heater was designed to melt and superheat the casting charge. The lower heater was designed to heat the mold. It also helps to maintain the superheat of the melt after it is cast, and helps to maintain the high thermal gradient at the solid-liquid interface that is required for DS. The upper heater is rated at 25 kVA and the bottom at 20 kVA. Furnace temperatures are measured by Type C thermocouples, controlled by a closed-loop Honeywell DCP 700 dual channel programmable controller, and monitored by a
Honeywell DPR 1000 multi-channel recorder. The power supply consists of isolated step-down transformers and silicon controlled rectifiers (SCR) power packs. An interlock turns off power to the heaters if the cooling water flow drops below a certain amount. The heaters are separated from the cold walls of the furnace by a multi-layer configuration of metallic (tungsten and molybdenum) radiation shields. A top access lid provides access to the hot zone for routine maintenance and inspection. The lower furnace chamber is used for loading, unloading, and cooling. A hinged rectangular door provides access to the lower chamber. A 3" (75 mm) diameter sight port, located on the door of the cooling chamber, and a small sight window on the top access lid provide for visual observation of the melt and the mold. Other access ports are also provided for control and monitoring thermocouples, a gas inlet, vacuum gages, and temperature pyrometers. Retractable baffles separate the hot and cold zones of the furnace, helping to maintain alloy superheat and to maximize the thermal gradient. A water-cooled copper chill plate is used to support the mold and promote the start of directional growth. The chill plate is supported by the pedestal travel system that allows the mold to be raised into the hot zone of the furnace and provides for withdrawal to the cooling chamber at a controlled rate. The pedestal travel system is of ball screw design and is powered by a DC stepper motor and electric gearbox-speed reduction. It is operated through a programmable controller. Casting can be performed either in low vacuum \(10^{-2} \text{ torr}, 1.33 \text{ Pa}\) or in inert gas. The evacuation system consists of a Leybold D30A dual-stage rotary vane mechanical pump, an electro-pneumatic roughing valve, DV-6M vacuum thermocouple gages, and a Granville-Phillips Series 307 vacuum gage controller. An inert gas inlet is provided and is operated through a solenoid valve. A pressure relief
valve controls the pressure of inert gas in the chamber and a graphite burst disk is used to protect against the possibility of chamber overpressure.

The operational capabilities of the as-received furnace were:

- Maximum operating temperatures:
  - Top heater: 2200 °C
  - Bottom heater: 2000 °C
- Operating pressure: $10^{-2}$ torr (1.33 Pa) low vacuum to 2 PSI (13.8 kPa) positive pressure inert gas
- Withdrawal speed: up to 30"/min (762 mm/min)

4.2 Modifications

During transportation of the facility to Carleton University and its subsequent re-assembly, some components were slightly damaged or deemed inappropriate for our purposes. The repairs consisted of adding molybdenum foil to the side shield assembly and installing an extra insulating ring composed of a zirconia ring and molybdenum foil. Sections of the original retractable baffles were replaced, as they did not accommodate our mold geometry. These repairs and changes are indicated on a section of the furnace chamber assembly drawing, Figure 4-5. In addition several modifications were performed to make the furnace more suitable for casting $\gamma$-TiAl alloys. These included the addition of a diffusion pump for higher vacuum capability and the modification of the inert gas system to allow for higher maximum positive pressure operation.
4.2.1 Heat Shields

During re-assembly it was noted that the side metallic heat shields were broken in several places near their bottom edges providing a direct view from the bottom heater element to the top shield cover, Figure 4-6. To protect the top shield cover from overheating, it was necessary to repair the side shield assembly. New strips of molybdenum foil (1” (25 mm) high, 0.0035” (0.09 mm) thick, and length to suit) were cut and added to the side shield assembly. The strips were added to the inside and the outside layers of the shield, forming two new layers. The strips were held in place with Type C thermocouple wire. The repaired assembly is shown in Figure 4-7. Since there were originally 4 or 5 layers of molybdenum foil, it was necessary to compensate for now having only two layers in some sections. Bonding a zirconia ring and molybdenum foil to the top shield cover provided added insulation. Strips of molybdenum foil were bonded to the inside of a zirconia ring using Cotronics Type 904 Ultra High Temperature Adhesive. The zirconia ring was ½” (13 mm) high, 3/8” (9.5 mm) thick, and 12” (305 mm) in outside diameter. The ring was then bonded to the top shield cover with the Cotronics 904 adhesive. The zirconia ring assembly is shown in Figure 4-8 and bonded to the top shield cover in Figure 4-9.

4.2.2 Retractable Baffles

The retractable baffle components were in poor condition and not compatible with the mold geometry. The mold pass-through was rectangular while for initial casting trials a round pass-through would be required. The original baffles are shown in Figure 4-10. New baffles were made using the same material: Type ZYFB zirconia board, ¼” (13 mm)
thick, from Zircar Fibrous Ceramics. A 10 ¼” (260 mm) diameter disk with a 1 ¼” (32 mm) diameter mold pass-through was cut from the board. Eight ¼” (6.4 mm) mounting holes were drilled to mount the baffle to the retractable skids. The baffle material was cut with a scalpel and the holes were drilled by hand using a ¼” (6.4 mm) bit. An 11/64” (4.4 mm) pass-through was also added for the bottom zone monitoring thermocouple. The finished baffle pieces are shown in Figure 4-11 and installed in Figure 4-12 and Figure 4-13.

4.2.3 High Vacuum Capability

Due to the high reactivity of titanium with oxygen a cleaner casting atmosphere was desired. The DS furnace as received was only capable of operation in a rough vacuum or small positive pressure of inert (argon) gas. Fortunately, the DS facility was designed for the addition of a diffusion pump and one was readily available on long-term loan from NRC. The addition of a diffusion pump allows for casting in the high vacuum range – approximately 10⁻⁵ torr (1.33 x 10⁻² Pa) – providing a much cleaner casting atmosphere. A diffusion pump mounting port, the internal control wiring, front panel controls, and PLC logic were originally supplied with the furnace. An Edwards CR160/700 cryo-cooled Diffstak Mk 2 was converted to electro-pneumatic operation, installed on the chamber, and wired to the control cabinet. To complete the conversion to high-vacuum operation, an ion gage tube (separated from the chamber with a manual isolation valve to allow the chamber to be operated at a high positive pressure), a foreline valve, a foreline trap, and assorted vacuum piping were installed. The components added are illustrated in Figure 4-14 and a schematic of the vacuum system is shown in Figure 4-15.
4.2.4 High Positive Pressure Capability

The volatility of aluminum at high temperatures and low pressures presents a problem in casting γ-TiAl alloys [15]. Alloy chemistry is difficult to control because of the evaporation of aluminum. Casting in a positive pressure of an inert gas, such as argon, has been shown to prevent the evaporation of aluminum. The original furnace was capable of operating at about 2 PSI (13.8 kPa) of positive pressure. It was believed that a higher positive pressure would be more beneficial. Consultation with Centorr Vacuum Industries determined that the furnace was capable of positive pressure to approximately 7 PSI (48.3 kPa). Therefore, the original 2 PSI (13.8 kPa) relief valve and 5 PSI (34.5 kPa) Zook burst disk were replaced by a 5 PSI (34.5 kPa) relief valve and a 5-7 PSI (34.5-48.3 kPa) adjustable spring-loaded relief valve, respectively, to allow for approximately 5 PSI (34.5 kPa) of positive pressure of inert gas during casting.
Figure 4-1: Carleton University DS facility – furnace chamber: (a) upper chamber, (b) control thermocouples, (c) retractable baffles, (d) lower chamber, (e) mechanical vacuum pump, (f) pedestal travel system, (g) power lines, and (h) cooling water lines.
Figure 4-2: Carleton University DS facility – control cabinet.

Figure 4-3: Schematic of the Carleton University DS facility furnace chamber.
Figure 4-4: Assembly drawing of the Carleton University DS facility furnace chamber.
Figure 4-5: Section of the furnace chamber assembly drawing illustrating where the repairs took place. 1: Addition of molybdenum foil to the side shield assembly. 2: Bonding of the zirconia ring with molybdenum foil to the top shield cover. 3: Replacement of the original retractable baffle components.

Figure 4-6: Damaged side shield assembly: (a) damaged shield assembly, (b) heater element, and (c) top furnace chamber.
Figure 4-7: Repaired side shield assembly.

Figure 4-8: Zirconia ring and molybdenum foil insulation assembly.

Figure 4-9: Zirconia ring and molybdenum foil assembly bonded to the top shield cover.
Figure 4-10: The original baffle components with a rectangular cutout.

Figure 4-11: New baffle components cut from the zirconia board.
Figure 4-12: New baffles as installed.

Figure 4-13: New baffles as installed, showing the retractable skids.
Figure 4-14: High vacuum components: (a) diffusion pump, (b) electro-pneumatic valve and actuator, (c) mounting flange, (d) ion gage tube and isolation valve, (e) foreline valve, (f) foreline trap, (g) vacuum piping, (h) mechanical pump, and (i) roughing valve.
Figure 4-15: Schematic of the vacuum system.
5 Nickel-base Alloy Casting Trials

The first goal of this project is the development of the equipment to process γ-TiAl alloys by DS. However, for several reasons initial DS casting trials were performed with a nickel-base alloy. Nickel-base alloys are more easily processed than γ-TiAl alloys: they have lower casting temperatures, there is less reaction between the melt and mold, and they have lower levels of volatile constituents. Also, since this facility was previously used to process nickel-base alloys the results of initial castings could be compared to the prior ones, providing an indication that the operational condition of the furnace is the same as before its relocation. It is also less costly to use a nickel base alloy and mold system to verify the operational status of the vacuum, heating, and withdrawal systems.

In all aspects, the facility is fully operational. The vacuum system can operate under rough or high vacuum conditions. The mechanical (rougning) pump can evacuate the furnace chamber to approximately $10^{-2}$ torr (1.33 Pa). With the diffusion (high vacuum) pump, vacuum levels of $3-4 \times 10^{-4}$ torr (~4.7 x 10^{-2} Pa) are consistently achieved. The heater elements and control system are fully operational. Temperature control at low temperatures, up to approximately 300°C, during heating is not very accurate. At higher temperatures, however, temperature control is quite accurate and the casting temperature
is maintained at a constant value throughout melting and withdrawal. The speed of the withdrawal mechanism is constant and controlled accurately.

Another important reason for initial casting trials was to determine the thermal characteristics of the furnace. The temperature gradients in the solidifying alloy were measured with thermocouples for several trials. These results will act as a baseline for estimating the temperature gradients in future DS γ-TiAl alloy casting tests. They also provide useful information for future modeling of the DS casting process. Casting trials with the nickel-base alloy are described below.

5.1 Experimental Procedure

5.1.1 Materials and Apparatus

A nickel-base ternary alloy was used for initial casting trials. It is the same alloy previously cast in this facility at Lehigh University [90]. Its composition, in weight percent, is 18.63 Cr, 16.18 Fe, 0.25 Al, 0.02 C, and balance Ni. The solidus and liquidus temperatures, respectively, are 1379°C and 1418°C. The alloy was supplied as a 3” (75 mm) diameter cast round bar and cut into slabs 5/8” (16 mm) thick. Molds were 8” (200 mm) long high-purity alumina tubes with 1” (25 mm) ID and 1 ¼” (32 mm) OD. Both melting and withdrawal were performed in these tubes. The DS furnace itself is described in Chapter 4.
5.1.2 Procedure

The casting process involves several steps. The first is the preparation of the alloy charge and mold, and loading into the furnace chamber. The second is the establishment of the protective casting atmosphere, involving chamber evacuation and argon back-fill operations. The chamber is then brought to casting temperature to melt the alloy charge within the mold. Finally, mold withdrawal is performed to solidify the charge. These steps are detailed below. Several different casting conditions were used and are summarized in Table 5-1.

The first step in the casting process involves preparing the alloy charge and the mold. The nickel-base alloy slabs were cut into smaller pieces with an abrasive cutting wheel and cleaned ultrasonically in acetone and then methanol. The charge was placed in the alumina mold. A 1" (25 mm) thick stainless steel disk was inserted in the bottom to raise the bottom of the charge into the hot zone and to protect the copper chill. For the first casting trial, as in previous ones at Lehigh, the cooling due to the chill was very effective and resulted in a large unmelted zone in the DS bar. For subsequent trials, a \( \frac{1}{2}'' \) (13 mm) thick zirconia disk was inserted between the copper chill and stainless steel disk to reduce the heat transfer. This resulted in a longer fully melted zone and DS portion in the cast bar. The mold and charge were placed on the copper chill plate and held in place with a stainless steel support disk. Figure 5-1 shows the mold in place on the chill plate. For several tests, a thermocouple (Type C) was used to provide an indication of the temperature profile of the melt during solidification. An alumina tube with one closed end protected the thermocouple, allowing it to be used repeatedly. The alumina tube was
cemented with a high temperature adhesive to a square piece of ½" (13 mm) thick zirconia board. This assembly sat on the top of the mold and supported the thermocouple in the melt, and is illustrated in Figure 5-2. The mold was then raised into the hot zone of the furnace. Its final position at the highest point of travel of the withdrawal mechanism placed the bottom of the alloy charge just above the top of the baffles. The retractable baffles were closed around the mold, leaving a small gap around the mold to allow for its movement during withdrawal.

The protective casting atmosphere was attained by chamber evacuation and back-fill operations. All casting trials were performed in approximately 5 PSI (34.5 kPa) of positive pressure flowing argon. For some trials only the mechanical pump was used to evacuate the chamber. To ensure a clean casting atmosphere, the chamber was evacuated and back-filled to approximately 10⁻² torr (1.33 Pa) and 2-4 PSI (13.8-27.6 kPa) positive pressure of argon, respectively, three times. The furnace was heated to casting temperature during the last evacuation. With the temperature of the chamber at approximately 1000°C the last argon back-fill was performed. The melting and withdrawal were then completed in flowing argon at 5 PSI (34.5 kPa) positive pressure. In other trials the chamber was evacuated with the high vacuum pump. The chamber was first evacuated with the mechanical pump, back-filled with argon, and re-evacuated with the mechanical pump. When the chamber vacuum reached 0.23 torr (30 Pa) evacuation was completed with the high vacuum pump. The furnace was again heated to casting temperature at this time, with the final argon back-fill at 1000°C.
Furnace heating to casting temperature was done in five stages: 5, 10, 20, 10, and 5 °C/min to 100, 500, 1460, 1500°C, and the casting temperature, respectively. The casting temperature was either 1520 or 1550°C – providing approximately 100 or 130°C of superheat. Slow initial heating rates were used to prevent thermal shock damage of the heating elements, shields, or the mold. The charge was held at the casting temperature for 15 minutes to ensure a homogeneous composition and temperature in the melt.

After the hold period the mold was withdrawn from the hot zone of the furnace into the cold zone, solidifying the charge. The casting temperature was maintained for the withdrawal. After the mold was completely withdrawn the furnace was cooled to below 100°C. The mold was then removed from the furnace.

To develop a better understanding of the casting process and the effects of casting parameters on the solidification microstructures of the alloy and the thermal characteristics of the furnace, the cast bars were examined by optical microscopy. The bars were first sectioned in half longitudinally by electro-discharge machining (EDM). Transverse sections were made near the top of the bar, near the middle, and at the location corresponding to the thermocouple position (when a thermocouple was used). All sections were polished and etched to reveal grain boundaries and the cellular/dendritic microstructure. The etchant was prepared by mixing 30% concentrated hydrogen peroxide (H₂O₂), hydrochloric acid (HCl), and distilled water (H₂O) in a 1:2:3 volume ratio. Submerging the sections for 5-15 seconds in the etchant revealed the grain boundaries and solidification morphologies. The area-intercept method (Equation 2.3)
was used to measure the average cell/dendrite spacing in the transverse sections. For the casting trials instrumented with a thermocouple in the melt, the temperature vs. time chart record was translated into a temperature vs. position plot. Temperature gradients in the liquid, solid, and mushy zones were determined from this plot. The results of these DS trials were compared to those of previous superalloy DS castings (c.f. Sections 2.2.3 and 2.2.3.1).

5.2 Results

5.2.1 Temperature Profiles and Gradients

Chart recorder plots of temperature vs. time for the thermocouple-instrumented tests are shown in Figure 5-3. The translated temperature vs. position plots are shown in Figure 5-4. Temperature gradients are measured as the slope of temperature vs. position and are tabulated in Table 5-2. Three separate gradients can be defined depending on the temperature at which they are calculated: (1) gradient in the liquid at the liquidus temperature – \( G_L \), (2) average gradient in the mushy-zone – \( G_{MZ} \), and (3) gradient in the solid at the solidus temperature – \( G_S \). Generally, for metallic alloys these gradients can differ significantly, especially at low growth rates [62]. This is apparent from the temperature vs. position plots in Figure 5-4 and the calculated temperature gradients listed in Table 5-2. For the purpose of comparing the results of these DS trials to previous superalloy DS castings (c.f. Sections 2.2.3 and 2.2.3.1) the gradient in the liquid at the liquidus temperature, \( G_L \), will be used.
5.2.2 Metallographic Analysis

An as-cast DS bar is shown in Figure 5-5. Polished and etched longitudinal sections are shown in Figure 5-6. They are arranged in order of increasing withdrawal rate, left to right. NA4, NA6, NA8, and NA10 were processed under identical casting conditions except for withdrawal rate. NA6NZ had no zirconia disk between the chill plate and the melt as evidenced by the unmelted zone and finer equiaxed and columnar grain sizes. NA6SH was processed at a higher superheat temperature. Zones of equiaxed grains are apparent in the section of each of the bars that was adjacent to the chill. The majority of the bars consists of columnar grain growth and closely resembles typical DS-processed superalloys.

Representative polished and etched transverse sections are shown in Figure 5-7 and Figure 5-8. This alloy solidified with cellular/dendritic morphology under the range of casting conditions used. As the growth rate increased, the solidification morphology changed from mostly cellular to maltese-cross-shaped cellular/dendritic, Figure 5-8. The PDAS measured from transverse sections for the various bars are shown in Table 5-2. These measurements were made near the top of the DS bars in the cases where thermocouples were not placed in the melt (NA6NZ, NA8, and NA10) and in the same cross section as the thermocouple for the thermocouple-instrumented bars (NA4, NA6, and NA6SH).
5.3 Discussion

The temperature vs. position plots in Figure 5-4 verify several important aspects of DS processing. Reducing the withdrawal rate, $V_l$, leads to a higher gradient and a smaller mushy zone. A reduction in $V_l$ from $4.22 \times 10^{-3}$ cm/s (NA6) to $2.82 \times 10^{-3}$ cm/s (NA4) resulted in an increase in $G_L$ from 32 to 40°C/cm and in $G_{MZ}$ from 49 to 67°C/cm. The mushy zone size decreased from a thickness of 8 mm to 5 mm. Increasing the casting temperature also leads to higher gradients and a smaller mushy zone. An increase from 1520°C (NA6) to 1550°C (NA6SH) resulted in an increase in $G_L$ from 32 to 53°C/cm and in $G_{MZ}$ from 49 to 66°C. The mushy zone size decreased from a thickness of 8 mm to 5 mm. Increasing $G_L$ with reducing $V_l$ and/or increasing casting temperature is as conventionally expected (c.f. Section 2.2.1). $V_l$ and the casting temperature also have an affect on the location of the mushy zone relative to the retractable baffle. Increasing $V_l$ or the control temperature moves the mushy zone further into the cold zone. This behaviour was illustrated in Figure 2-32 for increasing $V_l$ and verified in Figure 5-4. The mushy zone was closer to the top of the baffle for NA6SH than NA6 (increasing casting temperature) and for NA6 than NA4 (increasing $V_l$). This behaviour places a limit on the maximum possible withdrawal velocity, $V_{l_{\text{max}}}$, as given by Equations 2.7 and 2.8. As mentioned in Section 2.3.2, DS growth is only possible if the mushy zone (or solid-liquid interface) remains in the hot zone. Otherwise, radial heat transfer occurs and DS growth breaks down. This is starting to occur in the cast bar NA10 in Figure 5-6. $V_{l_{\text{max}}}$ can be calculated from Equation 2.8 using estimates of the physical properties of the nickel-base alloy from data in Ref. 52. Using $K_S \sim 25$ W/m°C, $G_S \sim 7500$°C/m, $\rho_S \sim 8000$kg/m³, and
\( L_r \sim 280 \text{ kJ/kg} \) yields \( V_{\text{max}} \) as \( 8.37 \times 10^{-3} \text{ cm/s} \), which is only slightly faster than the withdrawal rate of \( 7.06 \times 10^{-3} \text{ cm/s} \) used for NA10. The above results suggest that some degree of control over individual solidification parameters (\( V_1 \) and \( G_L \)) is seen to be possible. This is important in controlling solidification microstructures and ensuring that they are uniform throughout the cast component, as discussed in the following paragraph.

The combination of \( V_1 \) and \( G_L \) has a profound effect on the solidification behaviour of alloys. As mentioned in Section 2.2.3.1, \( V_1 \) and \( G_L \) have significant effects on the scale of the solidification structures found in a cast alloy. Higher cooling rates (the product of \( V_1 \) and \( G_L \)) lead to finer microstructures – as measured by DAS, Figure 2-24. The PDAS measurements for NA4, NA6, and NA6SH listed in Table 5-2 verifies this behaviour – as the cooling rate increased from NA4 to NA6SH the PDAS became finer. The solidification morphology of a cast alloy is also dependent on the combination of \( V_1 \) and \( G_L \), as discussed in Section 2.2.3 and illustrated in Figure 2-21 and Figure 2-23. This behaviour is apparent in Figure 5-8 for the current nickel-base alloy (NA6NZ, NA8, and NA10). As \( V_1 \) was increased, the solidification morphology changed from cylindrical cells (Figure 5-8 (a) and Figure 2-21 (c)) to flange/maltese cross-shaped cells (Figure 5-8 (c) Figure 2-21(d)). This occurs because a drop in \( G_L \) and/or an increase in \( V_1 \) result in instability of the liquid-solid growth front – recall from Equation 2.2 that a planar liquid-solid interface becomes unstable is the ratio \( G_L/V_1 \) drops below a certain value. The above observations demonstrate that by controlling \( V_1 \) and the casting temperature (and therefore \( G_L \)) some measure of control over the solidification morphology and scale can be obtained. Obviously, to obtain a uniform cast microstructure, it is desirable to ensure
that \( V_1 \) and \( G_L \) remain constant. The capability of the Carleton University DS furnace to accomplish this was verified by comparing the PDAS at several different cross sections for several of the cast nickel-alloy bars. The PDAS was found to remain constant (within 10\%) along the length of the bars. Therefore, \( V_1 \) and \( G_L \) remained constant throughout the entire casting process and steady-state condition existed for the duration of DS growth (excluding initial and final transients). The operational status of the furnace was also verified by comparing the PDAS of a nickel-base casting done at Carleton University to one performed at Lehigh University [90]. The PDAS of NA6NZ was 208 \( \mu \text{m} \) and the PDAS for the same alloy processed under the same casting conditions at Lehigh University was 230 \( \mu \text{m} \). These values are within 10\% of each other. The slight difference would be due to differences in mold/part geometry and different cooling water temperatures. These results demonstrate that the Carleton University furnace is capable of ensuring constant and repeatable casting parameters.

An important reason for the initial casting trials with the nickel-base alloy was to determine the thermal characteristics of the furnace. Specifically, it would be useful to estimate temperature gradients from PDAS in castings with no thermocouple instrumentation. This would be especially useful if the results could then be related to similar measurements in \( \gamma \)-TiAl castings – the reactivity of molten \( \gamma \)-TiAl alloys makes the placement of a thermocouple directly in the melt difficult and expensive. The measured PDAS and \( G_L \) for the nickel-base alloy castings NA4, NA6, and NA6SH were compared to those evaluated from the PDAS models, Figure 2-24 [57] and Figure 2-26/Equation 2.6 [56,62] presented in Section 2.2.3.1. These models will subsequently
be referred to as M1 and M2, respectively. For both models the measured $G_L$ and $V_t$ were used to predict a PDAS ($\lambda_{1M1}$ and $\lambda_{1M2}$) which was then compared to the PDAS measured from the transverse sections ($\lambda_{1avg}$). Conversely, for both models, the measured PDAS ($\lambda_{1avg}$) and $V_t$ were used to predict a gradient ($G_{LM1}$ and $G_{LM2}$), which was then compared to the gradient ($G_L$) that was measured by the thermocouple in the melt. The results of these comparisons are shown in Table 5-3 and Figure 5-9 and Figure 5-10.

From these results, it is apparent that the models M1 and M2 overestimate $\lambda_{1M1}$ and $\lambda_{1M2}$ predicted from the measured $G_L$ and $V_t$. They also overestimate predicted $G_{LM1}$ and $G_{LM2}$ from the measured PDAS ($\lambda_{1avg}$) and $V_t$. Model M1 is slightly better than model M2. This is especially apparent from Figure 5-9 and Figure 5-10.

The reason for the large discrepancy between model M2 and the experimental results is most likely due to the solidification morphology – the nickel-base alloy solidified with cellular or cellular/dendritic morphology and the model is based on well-defined dendritic growth morphology. The model M2 accurately predicts PDAS for the dendritic solidification morphologies found in superalloys. However, PDAS is known to drop off with a dendritic to cellular transition as $V_t$ is decreased at constant $G_L$ [62], Figure 5-11. This appears to be the case for this alloy – for the solidification parameters in question, a cellular morphology results, instead of a dendritic one. Therefore this model of PDAS, for dendritic growth, is not applicable in the case of this particular nickel-base alloy due to its cellular-cellular/dendritic growth morphology. The predictions of model M1 show better correlation to experimental results. This could be a result of the fact that the relationship between PDAS and $G_L$ and $V_t$ in the model M1 is a more general one. It was
derived as a curve fit for a relatively large number of different alloys that solidified under a range of different solidification morphologies (both cellular and dendritic). Due the fairly large scatter in these results, +/-30%, the current results seem to fit the model fairly well. However, this model cannot be reliably used to estimate $G_L$ from PDAS measurements, as evidenced by the results in Table 5-3. Therefore, to determine the thermal characteristics of the furnace, by estimating temperature gradients from PDAS in castings with no thermocouple instrumentation, a different alloy should be used. Under these growth conditions superalloys solidify with dendritic morphology. Therefore, to obtain a better understanding of the thermal characteristics of this furnace, a superalloy such as IN738 should be DS-processed instead of the nickel-base alloy that has been used to date.
<table>
<thead>
<tr>
<th>Specimen</th>
<th>Withdrawal Rate $V_1$, in/hr (cm/s)</th>
<th>Casting Temperature °C</th>
<th>Zirconia Disk?</th>
<th>Thermocouple?</th>
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<tr>
<td>NA4</td>
<td>4 (2.82 x 10^{-3})</td>
<td>1520</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>NA6NZ</td>
<td>6 (4.22 x 10^{-3})</td>
<td>1520</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>NA6</td>
<td>6 (4.22 x 10^{-3})</td>
<td>1520</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>NA6SH</td>
<td>6 (4.22 x 10^{-3})</td>
<td>1550</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>NA8</td>
<td>8 (5.64 x 10^{-3})</td>
<td>1520</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>NA10</td>
<td>10 (7.06 x 10^{-3})</td>
<td>1520</td>
<td>yes</td>
<td>no</td>
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</tbody>
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Table 5-1: Casting conditions used for initial testing with the nickel-base alloy.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Withdrawal Rate $V_1$, cm/s</th>
<th>Gradient liquid $G_L$ °C/cm</th>
<th>Gradient mushy zone $G_{MZ}$ °C/cm</th>
<th>Gradient solid $G_S$ °C/cm</th>
<th>Measured PDAS $\lambda_{lavg}$ μm</th>
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</thead>
<tbody>
<tr>
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<td>2.82 x 10^{-3}</td>
<td>40</td>
<td>67</td>
<td>78</td>
<td>242</td>
</tr>
<tr>
<td>NA6NZ</td>
<td>4.22 x 10^{-3}</td>
<td>~</td>
<td>~</td>
<td>~</td>
<td>208</td>
</tr>
<tr>
<td>NA6</td>
<td>4.22 x 10^{-3}</td>
<td>32</td>
<td>49</td>
<td>71</td>
<td>208</td>
</tr>
<tr>
<td>NA6SH</td>
<td>4.22 x 10^{-3}</td>
<td>53</td>
<td>66</td>
<td>82</td>
<td>183</td>
</tr>
<tr>
<td>NA8</td>
<td>5.64 x 10^{-3}</td>
<td>~</td>
<td>~</td>
<td>~</td>
<td>187</td>
</tr>
<tr>
<td>NA10</td>
<td>7.06 x 10^{-3}</td>
<td>~</td>
<td>~</td>
<td>~</td>
<td>173</td>
</tr>
</tbody>
</table>

Table 5-2: Gradients measured by thermocouples in the melt ($G_L$, $G_{MZ}$, and $G_S$) and the PDAS, $\lambda_{lavg}$, measured from transverse sections.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Withdrawal Rate $V_1$, cm/s</th>
<th>Measured PDAS $\lambda_{lavg}$ μm</th>
<th>Theoretical Gradient $G_{LMI}$ °C/cm</th>
<th>Theoretical Gradient $G_{LMI}$ °C/cm</th>
<th>Theoretical PDAS $\lambda_{lMI}$ μm</th>
<th>Theoretical PDAS $\lambda_{lMI}$ μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>NA4</td>
<td>2.82 x 10^{-3}</td>
<td>40</td>
<td>84</td>
<td>168</td>
<td>309</td>
<td>497</td>
</tr>
<tr>
<td>NA6</td>
<td>4.22 x 10^{-3}</td>
<td>32</td>
<td>89</td>
<td>186</td>
<td>291</td>
<td>503</td>
</tr>
<tr>
<td>NA6SH</td>
<td>4.22 x 10^{-3}</td>
<td>53</td>
<td>131</td>
<td>240</td>
<td>245</td>
<td>388</td>
</tr>
</tbody>
</table>

Table 5-3: Comparison of measured and theoretical gradients and PDAS calculated from models M1 [57] and M2 [56,62].

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Figure 5-1: View of the cooling chamber of the furnace with the mold held in place on the chill plate.

Figure 5-2: Assembly for thermocouple-instrumented casting trials: (a) zirconia board, (b) thermocouple protection tube, and (c) thermocouple.
Figure 5-3: Chart recorder plots for the thermocouple-instrumented trials, NA4, NA6, and NA6SH (top to bottom). The chart travel is from right to left. Trace 1 and 2 correspond, respectively, to the top and bottom zone control thermocouples (TC1 and TC2). Trace 3 corresponds to the thermocouple placed in the melt (TC3).
Figure 5-4: Temperature vs. position plots derived from the temperature vs. time plots for the thermocouples located in the melt for trials NA4, NA6, and NA6SH. The position is the distance of the thermocouple from the top of the retractable baffles (0 mm). Note the similarities of these plots to the one illustrated in Figure 2-32 for a binary alloy that solidifies at a discrete temperature. The liquidus temperature, $T_L$, is 1418°C and the solidus temperature, $T_S$, is 1379°C.
Figure 5-5: As-cast nickel alloy DS bar.

Figure 5-6: Polished and etched longitudinal sections. Casting conditions (withdrawal rates and casting temperatures) are given in Table 5-1. The growth direction is vertical.
Figure 5-7: Representative transverse sections under low magnification: (a) NA6NZ, (b) NA8, and (c) NA10.
Figure 5-8: Representative transverse sections under higher magnification: (a) NA6NZ, (b) NA8, and (c) NA10.

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Figure 5-9: Experimental data for NA4, NA6, and NA6SH plotted with the data from model M1 [57].

Figure 5-10: Experimental data for NA4, NA6, and NA6SH plotted with the data from model M2 [56,62].
Figure 5-11: Data from Trivedi [62] that shows the sudden decrease in PDAS with decreasing $V_l$, at a constant $G_l$, at the dendritic-cellular growth morphology transition.
6 γ-TiAl Casting Trials

The ultimate goal of this collaborative project is the development of γ-TiAl airfoil-shaped DS components. These components should have the proposed target microstructure to ensure a good balance of mechanical properties. However, before such components can be fabricated the processing/composition – microstructure – mechanical property relationships in these alloys must be determined. The first step in doing so is to choose a γ-TiAl composition suitable for initial DS processing trials. However, before this can be done, the method used to control the lamellar microstructure must be selected. The two available methods, described in Sections 2.4.3.1 and 2.4.3.2, are compositional control and use of a seed crystal. There are advantages and disadvantages to each that must be considered. The choice of method, selection of composition, and some preliminary experimental results from the first two DS casting trials are presented below.

6.1 Experimental Procedure

6.1.1 Materials and Apparatus

The mold material, from the initial project work, is high-purity Y$_2$O$_3$ [80]. The molds were 6 to 8" (150-200 mm) long with ¾" (19 mm) ID and 1" (25 mm) OD. The molds
were wrapped with graphite cloth held in place with Mo sheet and wire, as shown schematically in Figure 6-1. This was done to prevent leakage of the alloy charge if the integrity of the mold should fail during casting. The DS furnace itself is described in Chapter 4. The choice of alloy composition is discussed in the following two sections.

6.1.1.1 Method of Microstructural Control

The target microstructure can be achieved with the use of a “composite” seed, Section 2.4.3.2. A suitable seed composition can be found and can be used to control the microstructure in alloys with compositions different from the seed. Using a seed material in a production facility is, however, undesirable. A seed must be manufactured for each individual casting. In addition, a “composite” seed composed of many differently oriented seeds must be made to obtain the target microstructure. This is obviously not amenable to a production process as it is very time consuming and is not easily automated. Another potential problem occurs during the casting process if the molten charge is highly mixed, resulting in a loss of microstructural control. The other option is to choose an alloy composition so that the liquid fully transforms to the β phase upon solidification, resulting in an aligned microstructure similar to the target one. The final microstructure will have lamellar boundaries lying at angles ranging from 0° to 45° to the growth direction. While this is not as desirable as the target microstructure, it is an improvement over one that contains lamellae oriented perpendicularly to the growth direction. Controlling the lamellar orientation in this manner would be easier from a production point of view. This also makes the compositional choice more flexible as the only requirement is the full transformation to the β phase from the liquid. It was decided
that it would be best to control the lamellar orientation through compositional choice, as this method would be more amenable to the future full-scale manufacturing of components.

6.1.1.2 Determination of Alloying Additions

The choice of alloy composition is now quite flexible since the only requirement is the full transformation to the β phase directly from the liquid. There are two methods that can be employed to do so: the use of β stabilizing elements and/or a low aluminum content. From the review of γ-TiAl fundamentals given in Section 2.4.1 an alloy with a composition of Ti-46Al-2Mo-2Nb was chosen for initial DS casting trials. The low Al content will help promote β phase solidification and yield a fine lamellar spacing. Mo and Nb were added primarily as β stabilizers, but they also act as strengtheners and ductilizers. The master alloy was supplied by the Duriron Company as cast bars approximately ¾” (19 mm) in diameter and 8” (200 mm) in length.

6.1.2 Procedure

The casting procedure was similar to that used for the nickel-base alloy bars. The as received bars were cut to length to fit into the molds and the oxidation layer was ground off the surface. The bar was placed in the mold and then loaded into the furnace. A commercial purity titanium disk was place at the bottom to protect the copper chill plate. The high vacuum pump was used for all γ-TiAl castings. At 1000°C the final argon back-fill to 5 PSI (34.5 kPa) was performed. The same heating program was used to heat the furnace to 1520°C. Manual control was employed to raise the furnace temperature to
the final casting temperature of 1580 or 1600°C. After the charge was fully melted, it was held at casting temperature for 40 or 70 minutes to ensure a homogeneous composition and temperature of the melt. The withdrawal rate used for both trials was 4.7 in/hr (2 mm/min or 3.33 $\times$ 10$^{-3}$ cm/s).

The cast bars were examined by optical microscopy to determine the resultant solidification microstructure. They were first sectioned in half longitudinally by EDM and then polished and etched. The sections were ground and then manually polished using successively finer diamond pastes. The final polish was done with Fe$_2$O$_3$ powder suspended in water. Etching was done in a solution of 5 mL hydrochloric acid (HCl), 5 mL hydrofluoric acid (HF), and 90 mL distilled water (H$_2$O).

6.2 Results

6.2.1 Metallographic Analysis

The polished and etched longitudinal sections from the first two $\gamma$-TiAl DS casting trials are illustrated in Figure 6-2 (casting temperature of 1580°C, holding time of 40 minutes, and withdrawal rate of 2 mm/min – TAMN2-1) and Figure 6-3 (casting temperature of 1600°C, holding time of 70 minutes, and withdrawal rate of 2 mm/min – TAMN2-2). The resultant microstructures of these DS casting trials are illustrated in Figure 6-4 and Figure 6-5. These micrographs were taken in the regions marked by X in Figure 6-2 and Figure 6-3.
6.3 Discussion

The microstructures of TAMN2-1 and TAMN2-2 are qualitatively the same. A zone of equiaxed grains is visible at the bottom of the bar. Below this zone, a region of unmelted master alloy was present, but was removed before the metallographic preparation was done. The unmelted zone was quite long – accounting for approximately half the length of the master alloy bar. This was a result of the very effective cooling effect of the chill, mentioned in Section 5.1.2. The higher casting temperature and longer holding time for TAMN2-2 resulted in a longer DS portion in the bar. However, a good portion of the master alloy was still unmelted. A zirconia insulating disk placed under the titanium disk and the chill plate, as previously used for the nickel-base alloy casting trials, would increase the length of the DS portion. This would, however, result in rather large columnar grains, which are known to adversely affect ductility. Above the region of equiaxed grains is a zone of “radiant columnar” grains that appear to have grown in the radial direction. The top section of the bar consists of fully lamellar columnar grains. Unfortunately, as illustrated in Figure 6-4 and Figure 6-5, the lamellar orientation is not parallel to the growth direction. In most grains, the lamellae are oriented at 50 to 60° angles to the growth direction. However, a few grains just above the radiant columnar grains did have the desired parallel lamellar orientation. From these preliminary observations, the target microstructure was not obtained under the processing conditions used. Further casting trials to investigate the effects of the processing conditions on the alignment of the lamellar microstructure are required and are currently ongoing.
Figure 6-1: Crucible assembly for γ-TiAl DS casting trials.

Figure 6-2: Polished and etched longitudinal section of γ-TiAl ingot TAMN2-1. The growth direction is vertical. The micrograph in Figure 6-4 is taken from the region marked by X.
Figure 6-3: Polished and etched longitudinal section of $\gamma$-TiAl bar TAMN2-2. The growth direction is vertical. The micrograph in Figure 6-5 is taken from the region marked by X.

Figure 6-4: The lamellar microstructure of TAMN2-1 from the section marked by X in Figure 6-2. Growth direction is vertical.
Figure 6-5: The lamellar microstructure of TAMN2-2 from the section marked by X in Figure 6-3. Growth direction is vertical.
7 Summary and Conclusions

1. A dedicated facility for pilot-scale industrial DS processing of γ-TiAl components has been installed at Carleton University and is fully operational. Casting can be performed in high vacuum (3-4 x 10^-4 torr, \( \sim 4.7 \times 10^{-2} \) Pa) or at 5 PSI (34.5 kPa) positive pressure of flowing high purity argon. Casting temperatures of up to 1600°C have been used, while the maximum temperature capability is 2000°C.

2. Initial solidification trials with a nickel-base alloy were done to verify the operational status of the furnace and to determine the thermal characteristics of the furnace. Temperature gradients in the liquid, \( G_L \), in the range of 40°C/cm to 78°C/cm were measured. The casting parameters were shown to be controllable and repeatable and able to produce castings with a uniform microstructure throughout.

3. It was determined that the models for PDAS were not applicable for this particular nickel-base alloy due to the fact that it solidified with cellular-cellular/dendritic, not dendritic, morphology. Therefore, these models could not be reliably used to predict the temperature gradients from measured PDAS.

4. An alloy with a composition of Ti-46Al-2Mo-2Nb was chosen for initial γ-TiAl DS processing. To date, only two casting trials have been completed. Unfortunately, the target microstructure was not achieved under the processing conditions used.
7.1 Future Research

Now that the DS facility is operational and has been successfully used to cast γ-TiAl alloys the immediate future work would be the continuation of casting trials with the Ti-46Al-2Mo-2Nb alloy. This must be done to determine the effects of processing conditions on the alignment of the lamellar structure and to verify the suitability of the chosen alloy for DS processing. Ideally, a practical processing window should be identified that results in DS processed material with the target microstructure. Further work should also be directed to fine-tune the chosen alloy composition to make it more suitable for DS processing and to improve its mechanical properties. In addition, casting trials could also be performed with alloys of entirely different composition to determine the effect of ternary/quaternary additions on the solidification behaviour of γ-TiAl alloys. One such alloy has been identified and has a composition of Ti-47Al-2W-0.5Si. W is a strong β stabilizer and W and Si are known to improve the high temperature properties of these intermetallics, particularly the creep resistance. Extensive mechanical property data is available for the polycrystalline form of this alloy and would provide a useful comparison with results generated from the DS-processed material.

The choice of composition – Ti-46Al-2Mo-2Nb – should allow the role of the alloying additions of Mo and Nb to be more fully understood. Specifically the partitioning of Mo and Nb additions to the α₂ and γ phases, respectively, and the resultant changes on the domain boundaries and strength of the α₂ phase should be studied. It would also be interesting to determine if the quaternary alloy with both Mo and Nb additions would
have improved yield stress over a range of lamellar orientations, as mentioned in Section 2.4.1.4. The effects of these additions on environmental embrittlement of γ-TiAl alloys could also be investigated.

An important aspect that should be investigated is the use of finite element analysis (FEA) to model the furnace and casting to predict the effects of processing parameters on the solidification characteristics and resultant microstructures of γ-TiAl alloys. The importance of this is highlighted by the fact that, even for relatively simple alloys such as the nickel-base alloy used in this study, analytical models are not available or do not provide very accurate results. For complex alloys, such as the intermetallics, it would seem that FEA modeling would be necessary to relate the cast microstructures to the solidification parameters. This would facilitate the design of the DS casting process necessary to ensure the formation of the target microstructure and reduce the amount of slow and expensive experimental work that is required. A necessary component of such a modeling effort is calibrating the model with the actual casting parameters such as melt temperature, casting temperature, withdrawal rate, and position. The results of the thermocouple-instrumented nickel-base alloy casting trials will be useful for such a model calibration. Ideally, the temperature within the γ-TiAl alloy should be measured, but would be very expensive to do so due to the reactivity of the melt. If the temperature profiles for nickel-base and γ-TiAl alloys processed under the same conditions could be related, then an FEA model for a γ-TiAl alloy could be calibrated with data from a series of nickel-base alloy castings, significantly reducing the difficulty and cost. The possibility of doing this should be examined.
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