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**THE PROCESSING OF POWDER METALLURGICALLY
PRODUCED NEAR γ -TITANIUM ALUMINIDE**

by

JONATHAN C. BEDDOES, B.ENG., M.ENG.

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

Doctor of Philosophy

**Department of Mechanical and Aerospace Engineering
Ottawa-Carleton Institute for Mechanical and Aerospace Engineering**

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**THE PROCESSING OF POWDER METALLURGICALLY
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submitted by

JONATHAN C. BEDDOES, B.ENG., M.ENG.

in partial fulfilment of the requirements
for the degree of Doctor of Philosophy

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Aerospace Engineering

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1993

ABSTRACT

The technology of titanium aluminide intermetallics for aerospace applications is reviewed. Of primary interest are the properties and processing of titanium aluminides, concentrating on near γ -TiAl compositions. Inherent structural and deformation characteristics are described. The relationship between microstructure and processing is examined with emphasis placed on powder metallurgical, HIP'ing and hot deformation processes. Mechanical properties attainable with near γ -TiAl alloys are presented and the effect of ternary elements examined.

The microstructural characteristics of Ti-54%Al and Ti-48%Al-2%W were investigated to elucidate the microstructural evolution occurring during powder manufacture. Both powder compositions were consolidated by HIP'ing in the temperature range 1025 to 1250°C and the consolidated microstructures and mechanical properties evaluated. The HIP'ed compacts exhibit a non-uniform grain size associated with a varying volume fraction of dendrite regions in the powder. Tensile ductility is related to the homogeneity of deformation between prior dendrites and interdendritic regions. Tensile strength at room temperature and 550°C is dependent on the HIP'ed grain size, while at 850°C, strength is independent of grain size. The best combination of room temperature strength and ductility is 505 MPa and 1.3% elongation, obtained by HIP'ing at 1250°C.

The hot workability of Ti-48%Al-2%W was determined by isothermal constant strain rate forging simulations in the temperature range 900 to 1200°C and strain rate range 10^{-1} to 10^{-4} s $^{-1}$. Deformation and forgeability maps are presented for both 1050 and 1250°C HIP'ed material. The deformation map indicates the primary deformation mechanism occurring as a function of temperature and strain rate. At low temperatures and high strain rates deformation causes the development of a dislocation substructure. With increasing deformation temperature, compression causes dynamic recrystallization, which depending on the temperature/strain rate combination may occur concurrently with

superplastic flow. At the highest temperatures and slowest strain rates investigated, superplasticity predominates at low strains, followed by grain coarsening. The forgeability map illustrates the temperature/strain rate domain at which sound forgings are produced. Combining the deformation and forgeability maps indicates that crack free forgings are only produced by temperature/strain rate combinations at which superplastic flow contributes to the accommodation of strain.

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1. INTRODUCTION

The titanium aluminide intermetallics have recently become the subject of a wide body of metallurgical engineering research. The major reason for this focus is the potentially very favourable combination of low density and high temperature properties of these intermetallics (high specific strength). A good combination of these properties has always been important for gas turbine applications. In such applications, the motivation for development of higher temperature material capabilities is the increase in thermodynamic efficiency achievable with higher operating temperatures, while low density is beneficial in lowering the loads associated with the high rotational speeds of gas turbine components. These material requirements have prompted the major development effort directed toward nickel base superalloys during the past forty years. More recently, the initiative to build a hypersonic space plane has required the development of lightweight materials, for outer skin usage, that can withstand the raised temperatures associated with skin drag at hypersonic velocities.

In an intermetallic compound the ratio of the major elements is fixed, within a limited range, and the structure (or the lattice parameters) of the compound is specifically different from that of either of the base elements. Intermetallics are ordered because the A-B bonds have lower energy (greater strength) than the A-A or B-B bonds. Consequently, unlike atoms are favoured as first neighbours. The energy barrier of the order-disorder reaction is so high that order is retained to a considerable temperature (increasing entropy does not cause disorder). The strong A-B bonds and ordered structure result in a high activation energy for the diffusive mechanisms that often control many high temperature deformation properties (e.g. creep resistance, strength retention) ¹.

The two titanium aluminide intermetallics of particular interest are TiAl and Ti₃Al. These compounds possess several potentially useful properties, which are compared to titanium alloys and nickel superalloys in Table 1². It is evident that these compounds have several inherently useful characteristics: high modulus, low density and good oxidation resistance (due to high aluminum content). However a major problem has been low ductility below 600°C which causes workability and service problems. Many possible solutions to this problem have been investigated and these will be reviewed in subsequent sections.

1.1 TITANIUM ALUMINIDE APPLICATIONS

It is useful to briefly review the 'service' experience of TiAl compositions, as this leads to an appreciation of the state of the technology and the problems preventing further usage.

The gas turbine applications envisaged for TiAl and Ti₃Al are shown in Figure 1³. Considerable progress has been achieved in developing compositions that have a suitable balance of the properties required for some of the components shown in Figure 1, particularly for Ti₃Al, for which the low ductility problem has been dealt with more readily. A stator support ring of Ti₃Al was 43% lighter compared to the nickel base counterpart⁴, and a F100 engine compressor casing and divergent afterburner nozzle seals of Ti₃Al have been successfully engine tested⁵.

Development of TiAl compositions has not progressed to the same stage as Ti₃Al, primarily due to the low ductility problems. However, a General Electric T700 compressor case and 1st stage CF6 turbine blades have been investment cast from 'Ti-48%Al-2%Nb-2%Cr'^{6,7} and techniques for repair welding of hot isostatically pressed castings have been developed⁸. As well, F100 compressor blade preforms and 5th stage

* All compositions are in atomic percent unless otherwise indicated.

JT9D blades of TiAl+1%V have been successfully tested ⁹. However, a bladed PT6 rotor of this same alloy failed during spin pit testing at 70% of design speed during initial speed up ⁹. The failure occurred at blade roots and was caused by damage induced by grinding to improve the blade/rotor fit. This exemplifies the lack of defect tolerance of these alloys. Despite this, the technology required to investment cast and machine low pressure turbine blades from Ti-48%Al-2%V+7.5vol%TiB₂ has been developed ¹⁰.

Powder metallurgical (PM) techniques have also been successfully utilized to produce finished components ¹¹. Compressor vanes fabricated by hot die forging of TiAl compacts have been produced. The forging of TiAl PM compacts is the processing route investigated in this thesis and as such, will be discussed in greater detail in subsequent sections.

1.2 OBJECTIVES OF THIS THESIS RESEARCH

From the preceding section, it is evident that titanium aluminides can be expected to be used in future gas turbine engines for both stationary and rotating components. These applications require adequate elevated temperature strength, fracture toughness and ductility. These properties are dependent on alloy composition, microstructure and processing conditions. The influence of these parameters on mechanical properties is reviewed in the next chapter. However, very little information has been published that allows optimization of these material parameters for specific gas turbine applications. Hence, it is the purpose of this thesis work to investigate a potential processing route for a near γ -TiAl composition.

As will become clear in the following chapter, powder metallurgical techniques offer several advantages compared to ingot casting for many gas turbine applications. These advantages are related to the rapid solidification of metal powders, leading to a finer scale microstructure, affecting both grain size and dendritic segregation. Such microstructural refinement could improve workability during forging and contribute to

improved mechanical properties of TiAl components. Given these advantages, the potential processing route investigated involves the consolidation of procured TiAl powder into 100% dense compacts, followed by forging the compacted material.

This investigation of the consolidation and forging of TiAl required the completion of several major tasks.

1. To maximize the knowledge gained from the investigation, previous and current research and development work relevant to near γ -TiAl alloys was reviewed. This background information is summarized in chapter 2.
2. From the knowledge gained in the previous task, one TiAl composition was selected for further study. The specification of the selected composition and the selection rational will be summarized in chapter 3. Commercially produced powder of the selected composition was procured for this investigation.
3. Characterization of the microstructure of the procured powder was carried out, using optical and electron microscopy and energy dispersive X-ray analysis. This important step allows determination of the microstructural evolution during powder solidification and cooling. This knowledge is required in order to elucidate subsequent microstructure changes that occur during consolidation and forging.
4. The parameters required for consolidation of TiAl powder by hot isostatic pressing (HIP) to full density were established and resulting microstructures and mechanical properties (hardness, ambient and elevated temperature tensile properties) evaluated. This data allowed determination of the preferred consolidation conditions that can be used for the processing of PM compacts for forging evaluations.

5. The forging characteristics of γ -TiAl PM compacts (machined to 7mm diameter and 9 mm height) were determined. This was accomplished by isothermal constant strain rate compression testing using a 100 kN forging simulator. Such testing generated true stress - true strain data at elevated temperature for various strain rates. The compression behaviour was correlated with the microstructural changes occurring during compression. This allows determination of the metallurgical mechanisms controlling forging deformation and optimization of the forging parameters for the processing of components via a HIP plus forge route.

The results of tasks 3,4 and 5 are summarized and discussed in Chapters 4,5 and 6 respectively.

The primary significance of this thesis work is the demonstration that a near γ -TiAl intermetallic alloy can be successfully processed by hot forging of HIP'ed powder compacts. Moreover, the thesis work demonstrates this point by utilizing a TiAl+W composition, which as it will shown, possess inferior workability compared to many other near γ -TiAl alloys. Thus, this work defines a potential processing route for the fabrication of gas turbine parts from TiAl+W. Sound forgings have been produced after 100% compressive engineering strain, sufficient to allow fabrication of relatively complex near-net-shape parts. A thorough examination of the metallurgical processes occurring at all stages of the HIP plus forge processing route is presented, to elucidate the important attributes required of near γ -TiAl alloys for fabrication into gas turbine components.

2. BACKGROUND LITERATURE REVIEW

This chapter is intended to give an overview of the research and development on titanium aluminides to date. This will serve to put the thesis experimental work in perspective. This review and the thesis work concentrates on near γ -TiAl compositions, since these have the potential for superior high temperature properties compared to Ti_3Al . The first part of this literature review (up to section 2.4) describes the characteristics and properties of binary TiAl compositions. The influence of ternary elements is then discussed in section 2.5. Previous research on aspects of particular relevance to the experimental work of this thesis is reviewed more rigorously.

2.1 STRUCTURE

The structure of the two titanium aluminides TiAl and Ti_3Al differ: Ti_3Al being hexagonal and TiAl being a face centred slightly tetragonal structure. As a consequence of these structural differences the two intermetallics have considerably different microstructural and mechanical properties. However, the relationship between these two intermetallics is important, since in many compositions of interest, both structures exist as different phases in a single alloy.

TiAl (γ) has a stoichiometric composition of 37.3wt%Al (50at%Al) ¹². The face centred tetragonal structure of this compound is shown in Figure 2 ¹³. At 50%Al the structure is only slightly tetragonal, with a c/a ratio of 1.02. As is evident in Figure 2 the

aluminum and titanium atoms occupy alternate (002) planes, producing an ordered structure, with a Strukturbericht designation of $L1_0$.

From the provisional phase diagram of Figure 3 ¹⁴, TiAl is the equilibrium phase for a range of aluminum contents from about 49% to 66%, depending on the temperature. The nature of the phase diagram is described more completely in section 2.1.1. The ordered $L1_0$ structure is observed throughout this compositional range and depending on the prior processing, some second phases may be present ^{15,16}. Increasing the aluminum content above the stoichiometric ratio, causes substitution of the titanium by the aluminum in the superlattice (antisite substitution) ¹⁷. TiAl remains ordered right up to the onset of melting. Variation of the aluminum content slightly changes the volume of the unit cell, Figure 4 ¹⁷ (since titanium has a larger atomic diameter than aluminum). Decreasing the aluminum content decreases the tetragonality of the structure, Figure 5 ¹⁸, resulting in a structure closer to face centred cubic. The tetragonality is important when considering the deformation of TiAl, which is discussed in the section 2.2.

A range of compositions of particular interest is 46-54%Al. By altering the aluminum content in this range, single phase γ -TiAl is obtainable (>49%Al) or γ with a small volume fraction of α_2 -Ti₃Al is obtained (<49%Al).

Ti₃Al has a hexagonal structure of the DO_{19} type, which is illustrated in Figure 6 ¹³. Ti₃Al undergoes a disorder-order reaction at $\approx 1125^\circ\text{C}$ ^{16,19} and may contain antiphase boundaries at room temperature, depending on its thermal history. The α phase ordering to α_2 is generally very rapid with little apparent nucleation barrier ²⁰. Cooling Ti₃Al to $< 1125^\circ\text{C}$ causes supersaturation with aluminum ²¹ resulting in stacking faults on the Ti₃Al basal plane ²², which produce a local stacking sequence equivalent to the TiAl $L1_0$ structure. This situation is shown schematically in Figure 7 ²². This stacking sequence, repeated, can produce alternating laths of TiAl and Ti₃Al, with the orientation relationships, which are $(0001)_{\alpha_2} \parallel \{111\}_\gamma$ and $\langle 11\bar{2}0 \rangle_{\alpha_2} \parallel \langle 1\bar{1}0 \rangle_\gamma$ ²³.

A titanium aluminide composition of 48%Al typically contains TiAl and Ti₃Al phases in a lamellar structure when cast or cooled from the α region of the phase diagram of Figure 3. Subsequent heat treatment between 1100 and 1300°C will produce a 'duplex' structure with two types of grains, one having a γ -TiAl structure, while the other contains alternating laths of γ -TiAl and α_2 -Ti₃Al. The existence of the α_2 second phase limits grain growth considerably. The morphology of the microstructure of Ti-48%Al can be altered by heat treatment as seen in Figure 8²⁴. The selection of the heat treatment time and temperature (in the α + γ phase field) controls the ratio of lamellar grains to γ grains. Three types of microstructure are evident in Figure 8;

- 1) single phase equiaxed γ grains (Figure 8a),
- 2) a duplex structure of equiaxed γ grains and α_2 + γ lamellar grains (Figure 8b and 8c), and
- 3) a fully transformed α_2 + γ lamellar structure (Figure 8d).

In some ternary or quaternary compositions the α_2 + γ lamellar colonies may be intersected by secondary lamellar colonies which are oriented at some angle to the primary lamellae²⁵. The presence of the secondary lamellar colonies affects the fracture toughness as outlined in section 2.4.2. The effect of heat treatment on microstructure is discussed further in section 2.3.4. Clearly, the differences in grain structure of Figure 8 will have a marked effect on processing and mechanical properties, and this will be discussed further in later sections.

In TiAl the atomic bonding between the titanium and aluminum has a covalent character (the electronegativity of Ti is 1.54 and of Al is 1.61). The bonding of titanium and aluminum occurs by the interaction of electrons in the 'p' and 'd' orbitals of the aluminum and titanium respectively²⁶. Furthermore, the bonding is anisotropic because of the layering of the titanium and aluminum atoms in the superlattice. Consequently, in titanium containing planes there will be a strong component of d-d bonding, while in aluminum planes there will be a strong component of p-p bonding. In normal planes the p and d components of the bonding will vary with position in the superlattice. Intuitively, then, one would expect a strong directionality in the deformation behaviour and

mechanical properties. The covalent bond character also substantially increases the magnitude of the Peierls stress ²⁷, which can increase the sensitivity of mechanical properties to temperature. These features will be discussed further in subsequent sections.

The bonding character has been used ²⁶ to explain and predict the influence of various alloying elements on properties. Once again, this will be expanded upon later.

The electronegativity difference will also cause a difference in the charge densities surrounding the aluminum and titanium atoms ²⁸. The higher electronegativity of aluminum will give it a higher charge density and thus stronger bonding. This can have a significant effect on the nature of the fracture and will be highlighted in succeeding sections.

2.1.1 Ti-Al Phase Diagram

The exact nature of the phase diagram at near stoichiometric compositions has recently become the topic of considerable work. This work has focused on the identity of the phase in equilibrium with the γ -TiAl at temperatures between 1285 and 1480°C. The phase diagram of Figure 3, indicates this phase as β -Ti. However, an alternate version of the phase diagram suggests the existence of an α -Ti phase field at this temperature range. These two versions of the phase diagram are compared in Figure 9 ²⁹. The nature of this phase is important since it affects the microstructure (and consequently mechanical properties) of near stoichiometric compositions. The phases present in rapidly solidified Ti-46 to 56%Al alloys, which were heat treated at various temperatures, cooled and metallographically examined, are shown in Figure 10 ²⁹. These results suggest that α -Ti is the phase present with γ -TiAl. However, it has been postulated that the α -Ti phase nucleates because of undercooling of the melt and favourable nucleation kinetics ^{30,31}. A minimum of 100°C undercooling was estimated to be required to form metastable α -Ti ³⁰. However, hexagonal dendrites (α phase) have been observed in ingots 2 cm thick ²⁹, in which sufficient undercooling for the metastable formation of α would not be achieved.

Reference 32 has pointed out that metallographic determination of the phase diagram assumes that the high temperature microstructure is preserved during quenching (or cooling) to room temperature. But the high temperature β phase transforms (even when quenched) to α by nucleation and growth for compositions above 25%Al and martensitically for compositions below 25%Al. At all aluminum contents this α phase transforms to ordered α_2 at about 1125°C. The ordered phase can decompose into α_2 and γ phases because of the formation of stacking faults as outlined earlier. Since quenching from a single phase α region will result in the same microstructure, the high temperature phases cannot be determined metallographically.

The problem of phase transformations occurring during quenching has been overcome by the use of high temperature X-ray diffraction ³². In this technique samples of various compositions are heated in a stepwise fashion, with the X-ray diffraction spectra being determined at each temperature step. Using this technique, the partial phase diagram of Figure 11 ³³ was proposed, which is in close agreement with the solid lines of Figure 9 and is consistent with previous experimental observations ³⁰.

According to the phase diagram of Figure 11, for the compositional range 40 to 55%Al the phases present during equilibrium solidification and cooling can follow three different pathways ³²⁻³⁴. For compositions below 49%Al solidification begins with the formation of primary bcc β -Ti. In compositions with less than 45%Al, further cooling causes the β -Ti to become unstable and transform to hexagonal α -Ti. Compositions between 46 and 49%Al go through two peritectic reactions. The first involves hexagonal α surrounding the primary β dendrites and the concurrent transformation of the β dendrites to α . During the second peritectic, γ -TiAl forms in the interdendritic spaces, with the transformation of primary α to γ -TiAl. Cooling these compositions below 1125°C causes ordering of the α to α_2 -Ti₃Al, which as explained in the previous section, can decompose into α_2 and γ lamellae. Compositions between 49 and 55%Al form hexagonal dendrites of primary α , which on cooling are surrounded by and transform to γ -TiAl.

The investigations summarized in this section have successfully defined and explained the shape of the phase diagram, the commonly accepted version of which is shown in Figure 12¹³. However, in interpreting microstructures according to the phase diagram, two important points should be borne in mind³⁵:

1. Knowledge of interstitial contents at all stages is important. It is thought that up to 1500 ppm of oxygen does not affect the equilibrium phases present, but will alter the position of the phase boundaries. The important effect of carbon in this regard will be dealt with later.
2. Understanding of the equilibrium state is important. To achieve equilibrium may take up to 6 hours since the kinetics of several transformations involved (especially the $\alpha \rightarrow \gamma$) are very slow.

Unfortunately, few of the processing routes contemplated for titanium aluminides involve equilibrium cooling. The phase transformations occurring during non-equilibrium cooling are discussed in section 2.3.2.1. Obviously, however, without a clear determination of the equilibrium phase diagram, it would be nearly impossible to understand non-equilibrium reactions.

2.2 DEFORMATION

The γ -TiAl intermetallic has a large unit cell that is not isotropic. The resulting anisotropic deformation is one of the major factors leading to the brittleness of this compound. In common with other face centred structures the close packed plane of TiAl is {111}. However, because of its tetragonality and ordered structure the $\langle 110 \rangle$ slip directions of the {111} planes are not equivalent. Deformation by dislocations with a Burgers vector of $\frac{1}{2}\langle 110 \rangle^*$ will not destroy the ordered structure (see Figure 2) and this is the most favourable slip system (i.e. lowest energy)^{36,37} and is termed 'easy slip'³⁸.

* The $\langle hkl \rangle$ notation means that the h and k indices can assume all allowed values, but the l indice cannot change. As such, the dislocations in the set $\langle 110 \rangle$ are $[110]$, $[\bar{1}10]$, $[1\bar{1}0]$ and $[\bar{1}\bar{1}0]$.

Motion of dislocations with Burgers vectors of $\frac{1}{2}\langle 101 \rangle$ will destroy the ordered structure and create an antiphase domain. Passage of an identical dislocation will restore order. Hence, these dislocations must travel in pairs or as $\langle 101 \rangle$ superdislocations so that order is maintained ^{38,39}. Movement of superdislocations would intuitively appear to be a more difficult process (the Burgers vector is about twice that for $\frac{1}{2}\langle 110 \rangle$ dislocations) and so is often referred to as 'hard slip'. The $\frac{1}{2}\langle 112 \rangle$ superdislocation has also been observed ⁴⁰ and is the lowest energy superdislocation.

The various dislocations can undergo complex dissociation into partial slip vectors ^{38,41}. The dislocation $\frac{1}{2}[110]$ can divide into partials $1/6[211]$ and $1/6[12\bar{1}]$. Between these two partials is a region of stacking faults and an antiphase domain boundary. The superdislocations divide into partials one of which is $1/6\langle 112 \rangle$. This partial is pinned at temperatures below 630°C ³⁷, by faulted dipoles ⁴², and is also the twinning dislocation. Hence, the pinning of this partial dislocation renders the superdislocations immobile at low temperatures, as well as limiting twinning. Therefore, at room temperature deformation occurs predominantly by motion of $\frac{1}{2}\langle 110 \rangle$ easy slip ordinary dislocations, a limited amount of $\langle 112 \rangle[11\bar{1}]$ twinning and faults associated with the $1/6\langle 112 \rangle$ partial dislocation. The pinning of the $1/6\langle 112 \rangle$ twinning dislocation limits the extent of twinning at room temperature to not more than two twins in any one grain ³⁷.

Mobility of the various dislocations has also been explained by considering the anisotropic bonding present in TiAl ⁴². As discussed previously, in planes normal to those containing only titanium or aluminum atoms, the charge density varies with position. Consequently, any dislocations lying normal to planes containing only aluminum or titanium will experience a large Peierls stress and so be relatively immobile. Thus, dislocations with Burgers vectors parallel to $[112]$ (normal to the (112) plane which has only titanium atoms) will be immobile.

The lack of adequate slip systems (von Mises criteria) below 630°C results in a very limited ductility (less than 0.5%) exhibited by TiAl at room temperature. Additionally, the deformation will be very dependent on the direction of the applied load.

With increasing temperature above 630°C, the $\langle 101 \rangle$ superdislocations become increasingly active and the ductility is markedly increased due to these additional slip systems. The increasing mobility of the superdislocations with temperature above 630°C causes a brittle to ductile transition between 700 and 800°C, shown in Figure 13³⁷. The mobility of the superdislocations is brought about by two affects. Firstly, the partial dislocations $1/6\langle 112 \rangle$, which were pinned at low temperatures, become increasingly mobile with increasing temperature above 630°C, thus allowing superdislocation mobility. Since the $1/6\langle 112 \rangle$ partial dislocations are also the twinning dislocations, with their increased mobility above 630°C, $[112](11\bar{1})$ twinning becomes an active deformation mode. Secondly, the increased temperature provides sufficient thermal activation energy to reduce the effective Peierls stress of superdislocations⁴² It has been concluded³⁸ that the $1/6\langle 112 \rangle$ partial dislocations control two thirds of the dislocations in TiAl (the $\langle 101 \rangle$ superdislocations) and the twinning deformation.

The locking and consequent immobility of the superdislocations at room temperature could cause a strong Hall-Petch relationship for TiAl. The room temperature Hall-Petch plot is shown in Figure 14⁴³. The data of Figure 14 follows the relationship;

$$\sigma_y = \sigma_0 + k_y \cdot d^{-0.5} \quad \dots \dots (1)$$

where σ_y is the yield stress for a grain size d , and σ_0 is the yield stress in the absence of grain boundaries. The value of k_y determined from Figure 14 is $1.37 \text{ MN}\sqrt{\text{m}}$ ⁴³. A different k_y value of $1.1 \text{ MN}\sqrt{\text{m}}$ was obtained for wrought material⁴⁴. These relatively high values of k_y are usually associated with materials in which there is a strong locking of dislocations by some mechanism⁴², which is consistent with the dislocation interactions discussed. In contrast to the results of Figure 14, a second investigation of the Hall-Petch relationship for Ti-50%Al determined k_y to be $0.130 \text{ MN}\sqrt{\text{m}}$ ⁴⁵. This lower k_y value was

obtained by heat treating samples from the same ingot to attain different grain sizes, whereas the results of Figure 14 are compiled from several sources. Therefore, the lower value of k_y is probably a more accurate Hall-Petch parameter. Other explanations for the different k_y values are postulated as ⁴⁵:

1. interstitial impurities,
2. variance in starting dislocation density, which depends on prior processing history, and
3. crystallographic texture, which is yet to be investigated for TiAl.

In the plot of Figure 14 some data for two phase TiAl compounds have been included. The deformation behaviour of compositions just below the stoichiometric ratio that result in two phase structures (γ and lamellar $\gamma+\alpha_2$ grains) have been investigated ^{36,46-49}. As previously discussed a composition of Ti-48%Al results in a considerably finer grain size than single phase Ti-52%Al.

In two phase structures the predominant deformation mode at temperatures up to 870°C is by $\frac{1}{2}[110]$ easy slip ordinary dislocations and fault/twin formation ^{47,50}. Thus major differences between the deformation of single phase and two phase near γ -TiAl compositions are the propensity for twinning and the contribution of superdislocations to slip. In single phase TiAl no more than two twins in each grain were observed ³⁷, while in contrast, in two phase TiAl much more profuse twinning is observed. The increase in twinning deformation provides improved ductility for the Ti-48%Al composition compared to Ti-52%Al. The increased twinning is associated with the decrease in the aluminum content, which lowers the tetragonality and this diminishes the twin energy ⁵¹.

The different deformation behaviour of single and two phase alloys may also be influenced by the effect of impurity elements (C, O, N) which stabilize and segregate to the α_2 phase ⁵⁰. Removal of these interstitial elements from the γ phase decreases the degree of directionality of the Ti-Ti bonds, thus reducing the anisotropy of the Peierls stress ⁵². This Peierls stress reduction increases the $\frac{1}{2}[110]$ ordinary dislocation mobility.

As well, decreasing the interstitial content may increase dislocation mobility by reducing interstitial drag ⁴¹. Thus the $\frac{1}{2}[110]$ dislocations become more dominant as the volume fraction of α_2 is increased. A further factor contributing to slip in two phase alloys is the ability of the α_2/γ interfaces to act as sources of dislocations which can contribute to accommodating deformation ^{41,52}

2.3 PROCESSING & MICROSTRUCTURE

Many methods of processing TiAl intermetallics have been investigated to produce material in various forms. The processing of these intermetallics tends to be fairly complex, in large measure due to their low ductility and efforts to produce end products with satisfactory ductility. Ingot casting and powder metallurgical (PM) processes will be reviewed with the emphasis on the latter, since this is more relevant to this thesis work.

2.3.1 Ingot Casting

Ingot casting usually results in large grained structures, due to its slower solidification, with inferior properties ². Often these compounds are so lacking in ductility that cracking occurs during ingot cooling ^{53,54}. Even after hot isostatic pressing (HIP) and homogenizing for seven days at 1000°C a non-uniform grain size can still exist ⁵⁵. The properties of castings may be improved by isothermal forging at 1050°C (0.001/sec strain rate) with a uniform grain size of 25 μm after heat treatment at 1125°C and 70 μm after heat treatment at 1225°C ⁵⁶.

2.3.2 Powder Processing

The PM processing of titanium aluminides has received considerable attention since rapid solidification is capable of producing material with dendritic segregation on a much smaller scale than that occurring during casting ^{2,57}. PM processing also avoids

the fabrication problems resulting from the low ductility and brittleness of TiAl compounds.

Another advantage of PM processing is the ability to add second phases during compaction. This is utilized in the XD™ technique ⁵⁸, in which a dispersion hardening phase is introduced. In the case of TiAl the dispersed phase that has been investigated is TiB₂ ^{59,60}. Details of the process are proprietary. The properties of such compounds are discussed in section 2.5.7.

During solidification of powders the microstructural evolution will not follow the path outlined earlier in section 2.1.1. Before considering the various techniques for producing and compacting powders, the microstructural development during rapid solidification will be reviewed.

2.3.2.1 Microstructural Development During Rapid Solidification

During rapid solidification of powders considerable undercooling may occur and the peritectic reactions of the phase diagram can be partially avoided. Consequently, metastable phases can be produced, which depend on the composition and solidification rate (which in turn depend on the powder particle size).

Investigation of rapidly solidified Ti-Al compositions has produced the thermodynamic phase selection map of Figure 15 ³⁴. In Figure 15, the solid lines indicate the temperature or undercooling of the liquid required to nucleate solidification of the β -Ti, α -Ti or γ -TiAl phases. The dashed lines indicate the temperatures required to initiate the various solid-state transformations. In utilizing this thermodynamic map the nucleation and growth kinetics of each phase must be considered. This explains the following anomalies:

1. For <48%Al, β is the primary phase nucleated at all undercoolings, even though it is not always thermodynamically the most favourable phase. This is attributed to the more favourable kinetics of β phase nucleation.
2. The γ -TiAl phase should be the primary phase for large undercoolings of compositions of <47%Al and for small undercoolings of $\approx$ 54%Al. However, γ is rarely seen as a primary phase (at any composition) since its ordered structure results in sluggish nucleation and growth kinetics.

Thus for dual phase compositions (i.e. Ti-48%Al) β -Ti is the primary phase and transforms to α with further cooling. For single phase compositions (i.e. Ti-54%Al) α dendrites form during solidification and are then surrounded by interdendritic γ . For both compositions the α phase orders to α_2 -Ti₃Al at $\approx$ 1125°C. The α_2 decomposes to $\alpha_2+\gamma$ lamellae by the process outlined earlier, unless the cooling rate is sufficient to arrest this decomposition⁶¹. Hence, both solidification paths (β or α dendrites) lead to a room temperature microstructures of lamellar $\alpha_2+\gamma$ and γ regions.

The cooling rate (and undercooling) is determined, in large measure by the particle size, thus the volume fraction of dendrites and the $\alpha_2 \rightarrow \alpha_2+\gamma$ decomposition will depend on the particle size. This effect is seen in Table II³¹. The α_2 single phase plus dual phase $\alpha_2+\gamma$ represents the original dendritic regions, with the single phase γ -TiAl in the interdendritic spaces. The most notable trend is the increase in α_2 at the smaller particle sizes, due to a faster cooling rate and lack of time for the $\alpha_2 \rightarrow \alpha_2+\gamma$ decomposition.

In small powder particles a small fraction of martensitic α has also been observed⁶¹. This martensite again forms from primary β -Ti that was not surrounded by α during the $L+\beta \rightarrow \alpha$ peritectic. It is postulated that with high supercoolings (>250°C) powder may solidify segregate free as single phase β . The subsequent transformation to α then requires separate nucleation of α within this β , which is a difficult process, so the β transforms to α martensitically.

2.3.2.2 TiAl Powder Manufacture

Several of the techniques developed for production of metal powders have been successfully applied to TiAl. The four processes of particular interest are:

1. The 'rapid solidification rate' (RSR) centrifugal atomization process^{62,63}, in which a molten stream is poured onto a rapidly spinning disk, with molten droplets being ejected and solidifying in flight.
2. Equiatomic TiAl powders have been produced by mechanical alloying. This involves high energy ball milling of Al₃Ti and TiH₂ powders^{64,65} or elemental titanium and aluminum^{66,67}, to produce powder of 10 µm average diameter.
3. The plasma rotating electrode process (PREP) of Nuclear Metals⁶⁸, in which a helium plasma arc is used to melt the end of a rapidly rotating bar. The molten droplets spun off solidify in flight in a helium atmosphere.
4. The titanium gas atomization (TGA) process of Crucible Compaction Metals, which is described in further detail below, since the powder of this thesis investigation was produced by this technique.

The TGA process is described in References 69-72 and shown schematically in Figure 16. For the TGA process 27 kg. of metal is induction melted under vacuum in a water cooled copper crucible. In this manner a thin skull of the charge lines the crucible, thus eliminating the requirement for a refractory lining and the possibility of refractory contamination. Once molten the charge may be held at temperature to ensure proper homogenization, poured into the tundish and through a nozzle which directs the molten stream into a high pressure inert gas jet. The tundish and nozzle (which is made of tungsten or molybdenum) are also refractory free. The high pressure inert gas (argon or helium) discharges from an annular gas ring, impinges onto the molten metal stream, breaking it up into droplets which solidify in flight. The powder proceeds down the atomization tower and into the cyclone collector for further processing.

The powder produced by the TGA method is asserted to be nearly spherical and virtually free of segregation. A typical powder particle size distribution is shown in Figure 17 ⁶⁹. Note that 100% of the powder is less than 500 μm in size (-35 mesh). Notwithstanding the results of Figure 17, TGA produced γ -TiAl powder may require screening to -35 mesh size ⁷¹. As well, processing through a magnetic separator to remove magnetic particles which might be picked up during atomization may be necessary ⁷¹. Tap density of TGA powder is typically 65% and the flow rate according to ASTM B213 between 25 and 35 seconds for 50 grams of powder. The flow rate is a measure of how easily the powder will flow into a mould, with minimum values of 25 seconds usually considered acceptable.

According to Reference 70, changes in chemistry may occur during the powder manufacturing process. For example, during processing a TiAl-1%V compound only 0.1%V was lost, with aluminum content remaining unchanged. The typical interstitial content of TGA produced TiAl intermetallics is 200 ppm carbon, 50 ppm nitrogen, 20 ppm hydrogen and 800 ppm oxygen ⁶⁹. Of these interstitial elements only the oxygen changes during the TGA process, increasing by about 200 ppm.

2.3.2.3 Consolidation of TiAl Powders

The major disadvantage of the PM route is a higher oxygen content ^{57,61}, resulting from trace amounts of oxygen being trapped during the encapsulation step. Oxides on the surface of prior particle particles can locally impede grain growth during annealing by pinning of grain boundaries. TiAl PM compacts exhibit brittleness to higher temperatures than cast alloys and this has been attributed to higher oxide levels.

The hydrogen concentration in powders increases with exposure to hydrogen environments, especially above 500°C. Absorption of hydrogen by TiAl powders kinetically promotes the decomposition of the metastable α_2 phase to $\alpha_2 + \gamma$ ⁷⁴. However, it has been reported that for XDTM processed near γ -TiAl, no change in microstructure of

the alloy could be attributed to hydrogen ⁷⁵. Nevertheless, this effect combined with those of oxygen, highlights the importance of cleanliness during processing of TiAl powders.

Several interesting processes for consolidation of TiAl powders have been developed, which are briefly reviewed next. This is followed by a more detailed review of consolidation by hot isostatic pressing (HIP), since this is the compaction method used in this thesis investigation.

Titanium aluminide powder can be pressed into usable forms by extrusion, which requires temperatures in excess of 980°C and extrusion ratios of at least 5:1 to attain full density ⁶¹. A typical extrusion processing route can be as follows ¹⁷:

1. powder sieved, -30 mesh ($\leq 550 \mu\text{m}$ diameter) fraction retained,
2. can and press to a green density of 0.5 of full density,
3. evacuate to 1×10^{-5} mm Hg,
4. heat treat 1 hour at 1420°C, and
5. hot extrude with a 14-16:1 ratio.

This process resulted in a grain size of 25 μm (for a two phase composition).

Compaction of TiAl by hot pressing encapsulated powders by a pressurizing medium has been accomplished ⁷⁶. By using very high pressures (1.24 GPa) compaction can be accomplished in 60 seconds at 1200°C, avoiding the long exposure to high temperatures and the resultant microstructural changes that might occur. However, since the process is not truly isostatic, product shape is not always preserved.

TiAl powders have also been consolidated by shock wave synthesis ⁷⁷. In this process powder is packed into a stainless steel canister and a plane wave shock front is produced by projecting a steel plate against the canister. The heat generated by the shock wave causes localized heating and melting of the powder particle surfaces. Resolidification occurs quickly, due to heat conduction into the relatively cool particle interiors ⁷⁸. An advantage of this type of consolidation is the relatively cool bulk material

temperatures. This allows non-equilibrium microstructures, produced during rapid solidification to powder, to be retained. Indeed, single phase α_2 has been retained in a Ti-48%Al composition, for which the equilibrium phases are $\alpha_2 + \gamma$. Whether retention of the non-equilibrium phase is beneficial to the mechanical properties is yet to be determined.

2.3.2.3.1 HIP Consolidation of TiAl

2.3.2.3.1a Effect of HIP Parameters on Consolidation

During HIP processing a combination of temperature, isostatic pressure and time are utilized to achieve consolidation of powders to full density. However, the selection of HIP parameters should be such that undue coarsening of the rapidly solidified microstructure does not occur. During the HIP cycle, densification may occur by several processes, including; volume or boundary diffusion, deformation by creep or plastic yielding ^{79,80}. Consolidation shall occur by the superposition of these processes. However, depending on the alloy and HIP parameters one densification mechanism often predominates. For the case of γ -TiAl interparticle welding is hindered, because of the slow atomic diffusion inherent of ordered intermetallics ⁸¹.

The HIP parameters should be selected so that the final product is 100% dense. This has often involved a degree of trial and error. However, computer software that predicts the HIP'ed density has been developed ⁷⁹. The software algorithm assumes that densification occurs at a rate which is the sum of all possible mechanisms, based on a rate equation for each densification mechanism. For accurate predictions, it is thus necessary that the parameters controlling each potential consolidation mechanism be known or estimated. Depending on the alloy system of interest, this information may or may not be available. However, for the case of γ -TiAl the software predictions are in reasonable agreement with experiment ⁸⁰.

The influence of HIP parameters on densification has been studied with the use of an eddy current probe that monitors dimensional changes of the powder container

during HIP processing^{82,83}. Using such a probe the effect of temperature changes on density have been measured, Figure 18⁸³. Clearly, for full consolidation in one hour at 100 MPa, a temperature of 1000°C is required. The use of eddy current probes to develop preform shapes for HIP'ing of near-net shape parts has also been suggested.

The optimum time, temperature and pressure for HIP consolidation of γ -TiAl depends on the starting particle size distribution, morphology and composition. However, the general effects of time, temperature and pressure are similar for a wide range of γ -TiAl powders. Increasing the HIP time or temperature improves the density of the consolidated material, Figure 19⁸⁰. For a HIP time of 4 hours, it was observed that:

- a) at a HIP temperature of 800°C (1073K) interparticle bonding between powder particles were established,
- b) at 850°C (1123K) the remaining porosity was still interconnected, and
- c) at 900°C the remaining porosity was isolated.

In the case of Figure 19 full density is achieved by HIP'ing at 1066°C, 182 MPa and 4 hours.

The decrease in porosity density with increasing HIP time and temperature is shown in Figure 20⁸¹. The higher pore density near edges of the cross sections is attributable to the outflow of gas during HIP'ing. Full density is attained at HIP temperatures in excess of 1100°C. Furthermore, post HIP exposure to prolonged high temperatures (1200°C and 12 hours) leads to the reformation of large gas pores (up to 100 μ m) in material subjected to HIP at <1000°C or <140 MPa⁸¹. This demonstrates that HIP'ing is unable to entirely eliminate residual pores from consolidated material. Even subsequent to HIP'ing at 1200°C or 275 MPa, high temperature exposure caused the formation of fine gas pores.

A summary of the HIP cycles required to achieve full density for various γ -TiAl powders is listed in Table III^(68,73,80,81,84). The absolute minimum temperature and pressure necessary to achieve full density is 980°C and 98 MPa.

The HIP density predicted from computer algorithms is compared to experimental results in Figure 21⁴⁰. Given that various assumptions were made to determine the parameters controlling the rates of the HIP densification mechanisms, the predicted and experimental results are in good agreement. This is indicative of the usefulness of the prediction software, and removes some of the trial and error involved in optimizing HIP schedules. Note that for the γ -TiAl powders of Figure 21 the predicted dominant metallurgical mechanism during HIP consolidation is stage I & II creep deformation.

2.3.2.3.1b Effect of HIP on Microstructure

Many γ -TiAl compositions of interest are near the boundary of α/α_2 and γ in the phase diagram. Consequently, the phase changes occurring during HIP treatment are very dependent on the exact aluminum content. Also, the $\gamma/\alpha_2+\gamma$ phase boundary is not vertical. Accordingly, for some compositions (≈ 49 -50%Al) HIP consolidation may occur in the α , α_2 or γ phase fields. Moreover, the addition of ternary elements may alter the equilibrium phase boundaries and the kinetics of phase transformations. Owing to these factors, it is difficult to generalize regarding the phase changes that occur during HIP'ing.

Notwithstanding the above, a microstructural change occurring due to the extended time at elevated temperature of HIP treatments, is the transformation of the metastable α/α_2 phases into stable γ ⁴¹. This transformation occurs during the HIP cycle heating period at $\approx 800^\circ\text{C}$ and pressures of at least 100 MPa⁴⁵. It has been postulated⁴⁵ that the $\alpha_2 \rightarrow \gamma$ transformation enhances densification by the motion of dislocations passing through the α_2 phase to alter the stacking sequence to cause the transformation to γ . This dislocation motion is such that densification at powder particle interfaces is aided by local deformation. This reaction should be common to most rapidly solidified dual phase γ -TiAl compositions. As well, solute segregation due to the dendritic structure of the powder, is eliminated by homogenization during HIP treatment.

A very fine grain size results from HIP consolidation of γ -TiAl powders, Table III. HIP'ed grain sizes are consistently less than 30 μm . Note that ternary additions, such as vanadium, result in a considerably finer grain size ($<10 \mu\text{m}$)⁸¹. The effect of HIP temperature on grain size is illustrated in Figure 22⁸¹, with excessive grain growth occurring above 1100°C. The increasing grain size with temperature is due to the disordering reaction $\alpha_2 \rightarrow \gamma$. Additionally, if HIP'ing occurs in the single phase γ region then homogenization will eliminate the α/α_2 second phase and allow subsequent grain growth⁸⁰. HIP'ing at 850°C (of Ti-50.3%Al-1.8%Nb) caused no new grain formation⁸¹. The first new grains were observed at 900°C, growing to 12 and 28 μm average size with HIP'ing at 950°C and 1066°C respectively.

Increasing the HIP pressure from 140 to 275 MPa produced a finer grain size in Ti-50%Al-4%Nb, Figure 23⁸¹. This effect is probably due to more uniform dynamic recrystallization resulting from the higher HIP pressure.

2.3.3 Hot Workability

2.3.3.1 Isothermal Forging

During isothermal compression (650 to 1370°C) of near γ -TiAl flow softening occurs, either due to dynamic recrystallization at slow strain rates (10^{-5} to 10^{-2} sec^{-1})^{86,87} or deformation heating at faster strain rates (0.1 to 10 sec^{-1})⁸⁸. The former strain rate range is typical of forging operations and for TiAl generates true stress strain curves of the type shown in Figure 24⁸⁶. The constitutive equation for isothermal deformation of this nature has been suggested as⁸⁹:

$$\dot{\epsilon} = A' (\sinh \alpha \sigma)^{n'} \exp(-Q/RT) \quad \dots \dots (2),$$

where Q is the activation energy, T is the absolute temperature, R is the universal gas constant, and A' , n' and α are material constants. At low stresses equation (2) simplifies to:

$$\dot{\epsilon} = A \sigma^n \exp(-Q/RT) \quad \dots\dots (3) .$$

Usually this equation is rewritten to incorporate the temperature compensated strain rate or Zener-Hollomon parameter, Z, as;

$$Z = \dot{\epsilon} \exp (Q/RT) \quad \dots\dots (4) .$$

By plotting $\ln \dot{\epsilon}$ versus $1/T$ for a constant true stress (usually constant peak true stress), the activation energy can be obtained from the slope ⁹⁰ as:

$$Q_{\sigma} = -R \left[\frac{\partial \ln \dot{\epsilon}}{\partial (1/T)} \right]_{\sigma = \text{constant}} \quad \dots\dots (5) .$$

In this manner, Q_{σ} is the activation energy corresponding to the selected stress level for a range of strain rates. Note however from equations 3 and 4 that;

$$Z = A \sigma^n \quad \dots\dots (6) .$$

Therefore, the activation energy, Q_{σ} , is only valid over the strain rate range for which equation 6 is valid, that is a plot of $\log \sigma$ against $\log Z$ is linear. Figure 25 illustrates this relationship for two near γ -TiAl compositions isothermally compressed at strain rates of 10^{-2} to 10^{-5} s^{-1} ^{86,91}.

Activation energies (Q_{σ}) of 308 to 426 kJ/mole have been reported for various near γ -TiAl alloys ^{91-93,96}. These activation energies are in the same range as those for creep deformation (see section 2.4.3) during which dynamic recrystallization also occurs. Indeed, during slow strain rate isothermal forging the onset of dynamic recrystallization occurs at the peak true stress ^{86,94-95}. Thus, the dynamically recrystallized grain size is a function of peak true stress, see Figure 26 ⁸⁶. By using Figures 25 and 26 the grain size after deformation can be predicted from the compression strain rate and temperature. Thus, it may be that dynamic recrystallization is the mechanism controlling both creep and compression forging deformation.

Isothermal forging of the lamellar $\alpha_2+\gamma$ microstructure results in activation energies of up to 672 kJ/mole⁹⁷ and less workability. It is suggested that these effects are due to the strong anisotropy of plasticity caused by the lamellar structure⁹⁸.

The activation energy for isothermal deformation may also be expressed for constant true strain as⁹⁸:

$$Q_\epsilon = \frac{R}{m} \cdot \left[\frac{\partial \ln \sigma}{\partial (1/T)} \right]_{\epsilon = \text{constant}} \quad \dots \dots (7) ,$$

Where m is the strain rate sensitivity exponent of the equation:

$$\sigma = C \dot{\epsilon}^m \quad \dots \dots (8a)$$

$$\text{so } m = \left[\frac{\partial \log \sigma}{\partial \log \dot{\epsilon}} \right]_{T, P = \text{constant}} \quad \dots \dots (8b) ,$$

which is commonly used to relate true stress and strain rate. The derivation of this activation energy is given in Appendix A. Note that for Q_ϵ the effect of strain rate is introduced through the strain rate exponent, m . Thus, in contrast to Q_σ , both the strain and strain rate influence the value of Q_ϵ . This activation energy to a selected constant true strain level has not been used to evaluate data for near γ -TiAl alloys. However, constitutive relationships for Ti-6242 (Ti-6%Al-2%Sn-4%Zr-2%Mo) have been determined using Q_ϵ ⁹⁹.

The strain rate sensitivity exponent can also be regarded as a power partitioning index¹⁰⁰, for distributing the forging work between creation of adiabatic heat and energy required for microstructural changes. Adiabatic heating is typically only significant at strain rates of greater than 10^{-1} s^{-1} . An efficiency parameter:

$$\eta = \frac{2m}{m+1} \quad \dots \dots (9) ,$$

can be defined¹⁰⁰ as a dimensionless parameter describing how well the workpiece dissipates power by structural changes.

Interactions between structural evolution and flow stress can be determined by relating the dynamically recrystallized grain size, D , and peak flow stress according to:

$$\sigma_p = M \cdot D^{-q} \quad \dots\dots (10),$$

where M and q are empirical constants ⁸⁶. For Ti-50%Al the grain size and peak stress is related as seen in Figure 27. For peak stresses >300 MPa, $q=0.5$ corresponding to strengthening by a Hall-Petch mechanism. At lower peak stresses $q=1.0$, indicating an enhanced rate of recovery at higher temperatures preventing the occurrence of dislocation pile-ups necessary for the Hall-Petch mechanism to be operative ⁸⁶. A similar result is seen in Figure 28, in which the activation energy for compressive deformation changes at a Zener-Hollomon value of $\approx 10^{14}$ ⁸⁶. At lower Z values the deformation structure consists of fine equiaxed recrystallized grains, while at high Z values few recrystallized regions exist. Thus, at low Z values dynamic recrystallization is predominant and at high Z stress relaxation occurs primarily due to dynamic recovery.

Complicating the above analysis is that dynamic recrystallization caused by forging in the $\alpha_2+\gamma$ phase field may not be complete, with coarse aluminum rich grains and parts of the initial $\alpha_2+\gamma$ lamellar structure retained after forging 80% ⁹⁷. Subsequent heat treatment in the $\alpha+\gamma$ region completes the recrystallization process, but results in an inhomogeneous α_2 distribution. The consequence of this process is early fracture initiation in coarse grained regions. As such, a suggested procedure is to forge in the $\alpha+\gamma$ phase field, producing a homogeneous fine distribution of the α_2 phase, followed by forging in the $\alpha_2+\gamma$ region to produce a more fine grained structure and improved mechanical properties ⁹⁷.

For Ti-48%Al-2%Cr-2%Nb, the occurrence of cracking during isothermal compression testing has been used to generate the deformation map of Figure 29 ⁹¹. Deformation in the poor workability regime results in cracking primarily along the γ/α_2 interfaces in lamellar regions. The deformed microstructure contained bands (15 to 25 μm wide) of recrystallized γ between regions of heavily deformed material, as well as thin strips of

partially recrystallized material between coarse recrystallized γ grains. It was postulated that the inhomogeneous deformation causes shear bands, which promote localized recrystallization, thus accommodating higher strains ⁹¹.

2.3.3.2 Superplasticity

Several sources of superplasticity for metals have been identified, however of particular interest is micrograin superplasticity. The main requirements for this phenomenon are a small grain size (typically $<10\text{ }\mu\text{m}$), a temperature in excess of $0.4T_m$ (in absolute degrees) and a controlled strain rate ¹⁰¹. Superplasticity is characterized by a high strain rate sensitivity (m) over some range of strain rates. Thus, in the superplastic range the flow stress is strongly dependent on the applied strain rate, Figure 30 ¹⁰², with $m \geq 0.30$. Usually, decreasing the grain size or increasing the temperature shifts the superplastic region to higher strain rates. Micrograin superplastic deformation occurs by grain boundary sliding, with an accommodation process occurring at the triple points. This accommodation process can occur by diffusion, dislocation motion, grain boundary migration or grain rotation and rearrangement.

Binary alloys of Ti-50%Al ¹⁰³ and Ti-42.7%Al ¹⁰⁴ produced from powder and thermomechanically processed by hot pressing or forging at temperatures of $\sim 1000^\circ\text{C}$ exhibit tensile elongations of $\approx 250\%$ when tested at $\approx 1025^\circ\text{C}$ and an initial strain rate of $2.5\text{--}8.3 \times 10^{-4}\text{ sec}^{-1}$. A maximum strain rate sensitivity coefficient (m) for the Hollomon equation (equation 8a) of 0.43 was observed and grains as large as $40\text{ }\mu\text{m}$ existed however the average grain size was $<5\text{ }\mu\text{m}$ ¹⁰³. The activation energy (Q_a) for superplastic deformation of Ti-42.7%Al is 380 kJ/mole. This is within the range of activation energies for creep deformation (see section 2.4.3) and suggests a similar metallurgical mechanism may control both superplasticity and creep. Superplasticity has also been observed with Ti-47%Al-3%Cr ^{105,106}. In this material, which was cast, homogenized (96 hr/ 1050°C) and forged 60%, tensile elongations of 450% were obtained at 1200°C and an initial strain rate of $5.4 \times 10^{-4}\text{ sec}^{-1}$. An m value of 0.57 was measured and the grain size was $\sim 18\text{ }\mu\text{m}$.

During superplastic deformation dynamic recrystallization occurs. More importantly, in the binary alloy - which contained a heterogeneous grain size after forging - it was noted that large grained regions recrystallized and fine grained regions coarsened during superplastic flow ¹⁰³. In this alloy superplastic behaviour was related to the ability of the grain boundaries to 'absorb' dislocations and accommodate dislocation motion, thus acting as 'channels' for plastic flow. As such, the grain boundary structure is important. A higher portion of arbitrary boundaries, as opposed to twin boundaries, enhances superplastic flow because of their greater ability to accommodate dislocations.

In the ternary alloy β -Ti₂AlCr was present after forging at triple points. During superplastic flow, the β phase spreads out along grain boundaries and has a higher dislocation density than γ and dynamically recrystallized more easily than γ . Hence, it was concluded that β phase promotes superplasticity by accommodating deformation and allowing grain boundary sliding ¹⁰⁵.

Superplasticity can also occur in binary TiAl with a sub-micron grain size. Such behaviour has been observed in hot pressed mechanically alloyed powder ⁶⁷, as well as cast and thermomechanically processed material ¹⁰⁷. Superplastic deformation in such material was attributed to a mechanism of grain boundary sliding activated at temperatures as low as 650°C ¹⁰⁷.

2.3.4 Heat Treatment

As is evident from Figure 8, the microstructure can be altered substantially by heat treatment. Figure 31 illustrates the heat treatment profiles that can be applied to cast and forged ^{108,109} or PM material hot pressed and extruded ²⁴. In scheme I and II of Figure 31 the solution heat treatment ranges in time from 0.5 to 5 hours and the temperature applied corresponds to the $\alpha+\gamma$ or α phase fields ¹⁰⁶. The higher temperature step ($T \approx T_\alpha$) serves to modify the grain size and volume fraction of α , γ or lamellar structure. The lower temperature aging treatment and scheme III is used to stabilize the microstructure prior

to service. The purpose of a stabilization heat treatment is to ensure that at the intended service temperature, the initial microstructure is in a near equilibrium state. In this way, microstructural changes will not occur during extended service exposure to elevated temperatures.

The solution heat treatment temperature controls both the grain size and volume fraction of the lamellar structure present. Solution treatments at temperatures just above the α_2 disordering temperature ($>1125^\circ\text{C}$) result in a large volume fraction of coarse γ grains, along with fine α_2 or lamellar grains ^{24,109}. Such a microstructure is in agreement with the phase diagram of Figure 12, in which a small fraction of α exists at temperatures just above 1125°C . Solution treatment of fine grained PM produced materials within the $\alpha+\gamma$ or α phase fields causes an increasing amount of lamellar structure and a larger grain size ^{24,110}, Figure 32. The former trend is due to the larger equilibrium proportion of α with increasing temperature in the $\alpha+\gamma$ phase field (see Figure 12). The α grains decompose to $\alpha_2+\gamma$ lamellae upon cooling to below 1125°C as outlined in section 2.1. These microstructural features are evident in Figure 8 and the respective mechanical properties are discussed in section 2.4.1. Heat treatment at temperatures $>T_a$ causes complete transformation to the α phase, which on cooling decomposes to $\alpha_2+\gamma$ lamellae (see Figure 8d). At such heat treatment temperatures rapid grain coarsening occurs, which in cast and forged near γ -TiAl can double the grain size in only 2 minutes at 1430°C ¹¹¹.

Interestingly, in Figure 32 ¹¹⁰ the volume fraction of the lamellar structure does not increase with the solution treatment time, however the grain size continues to coarsen. This indicates that the $\gamma\rightarrow\alpha$ transformation during heat treatment is relatively rapid. The rapid kinetics of this reaction may be expected since it involves transformation from an ordered to disordered state. As mentioned in section 2.1.1 the kinetics of the reverse reaction ($\alpha\rightarrow\gamma$) are very slow requiring up to 6 hours to reach equilibrium ³⁵.

The heat treatment schemes of Figure 31 can result in the lamellar microstructure via three different transformation sequences in cast and forged near γ -TiAl ¹⁰⁹. The first

type of lamellar structure is caused by slow cooling from the α phase, corresponding to the reaction:

in which γ plates precipitate within the disordered α phase (γ_p), subsequently forming the lamellar (lam) structure ¹¹². It was observed that the lamellar spacing from this type of reaction is usually $<0.2 \mu\text{m}$, and the α_2/γ lamellae have the $(0001)_{\alpha_2} \parallel (111)_{\gamma}$ or $[2\bar{1}\bar{1}0]_{\alpha_2} \parallel [110]_{\gamma}$ crystallographic relationship ¹¹³.

A second type of lamellar structure can be formed by solution heat treatment at about 1300°C , followed by relatively faster cooling ($\approx 80^\circ\text{C}/\text{minute}$ for cast and forged material) ¹¹². The faster cooling delays the precipitation of γ to below the α ordering temperature, conforming to the following reaction:

where γ_{pt} and γ_p refer to γ precipitates and plates within the α matrix respectively. This α_2 decomposition reaction requires the formation of stacking faults and results in the α_2/γ crystallographic relationships discussed in section 2.1. When the solution heat treatment temperature is lowered to just above the α ordering temperature (e.g. $1150\text{-}1225^\circ\text{C}$) this reaction is infrequent due to the low aluminum content of the α , which reduces the driving force for γ formation. The lamellar structures resulting from the transformations of equations 11 and 12 are very similar and it is not always clear which of the two reactions may have occurred.

Another lamellar structure composed of coarse α_2 plates and γ plates of the same crystallographic orientation can be formed by the sequence:

In this reaction the α_2 particles (α_2^P) within a γ matrix (γ_m) develop into a plate morphology (α_P) during isothermal heat treatment in the $\alpha+\gamma$ phase field, thus forming a lamellar $\alpha+\gamma$ structure. On cooling this transforms to an $\alpha_2+\gamma$ structure, via the $\alpha \rightarrow \alpha_2$ ordering transformation. The resulting γ regions all have the same crystallographic orientation ¹¹².

2.4 MECHANICAL PROPERTIES

The two major mechanical property issues regarding near γ -TiAl compositions are low ductility at room temperature and poor impact resistance. Both these properties require improvement before ‘damage tolerant’ applications can be considered. The ductility and impact properties are reviewed in the first two parts of this section. Due to these two problems, less work has been undertaken on the creep and fatigue resistance of near γ -TiAl, however the preliminary work in these areas is reviewed in the third and fourth sections. The last section is a review of the effects of HIP’ing on mechanical properties, which is important to this thesis work.

2.4.1 Tensile Properties

Considerable effort has been directed toward developing acceptable room temperature ductility in TiAl. From this work the highest room temperature ductility reported to date for a binary composition is 2.1% for Ti-48%Al ¹¹³ and 3.5% for ternary alloys ¹¹⁴. Several factors associated with improved ductility are

- 1) decrease in lattice tetragonality (c/a reduced),
- 2) decrease in unit cell volume,
- 3) decrease in impurity content,

- 4) presence of twins (additional deformation mode), and
- 5) a duplex microstructure of γ grains and $\alpha_2 + \gamma$ lamellar grains.

All of these factors (except 3) are influenced to some extent by the aluminum content. The relationship between the tensile properties and aluminum content is illustrated in Figure 33¹¹⁵. However, the contribution of each of the factors given above to the improved tensile properties for lower aluminum contents is not completely clear, partly because the strength of an alloy may depend on the characteristics of both phases. Thus correlations between strength and ductility and parameters such as c/a ratio are not consistent²⁰.

The change in lattice tetragonality due to the aluminum content is shown in Figure 5. The lower tetragonality will make the structure closer to face centred cubic and so all $\langle 110 \rangle$ slip directions may become more equally active. The improved ductility of Ti-48%Al has been correlated with mobile easy slip ordinary $\frac{1}{2}[110]$ dislocations and $\{111\}$ twins⁴⁷. In contrast, Ti-52%Al exhibited extended superdislocations and many sessile fault dipoles.

As outlined in section 2.1 reducing the aluminum content from 52% to 48% results in a duplex microstructure with enhanced ductility to at least 800°C as seen in Figure 33. Furthermore, the brittle to ductile transition temperature is considerably lower for the duplex Ti-48%Al composition. As seen in Figure 8, the microstructure may be altered by heat treatment. The tensile properties of each heat treated condition will be influenced by the deformation behaviour, which in turn is determined by the microstructure. The relevant duplex microstructural parameters are¹¹⁶;

- 1) thickness of TiAl lamellae,
- 2) size of domains in TiAl lamellae,
- 3) volume fraction, distribution, orientation and thickness of α_2 -Ti₃Al lamellae,
- 4) size of lamellar grains and γ -TiAl grains, and
- 5) volume fraction of each type of grain.

A decrease in the thickness of TiAl lamellae increases not only strength but also ductility, because the uniformity of deformation increases. Finer domains within TiAl lamellae improve ductility since deformation is more isotropic. Lamellae interfaces also improve ductility since they lead to crack bifurcation during fracture ¹¹⁷. In contrast, sufficiently thick Ti₃Al lamellae obstruct slip and shorten the length of slip bands, thus playing the same role as grain boundaries ¹¹⁸. In ternary alloys containing chromium, coarsening of the lamellar structure reportedly improves ductility ¹¹⁹ through several effects. Elimination of α_2 laths causes single phase γ regions within the previously α regions which can deform in the same manner as primary γ without hinderence of the α_2 laths. Also, the lower aluminum content of the γ coarsened from the primary α will have lower stacking fault energy ¹¹⁹ and the formation of thermal twins may act as sources of mechanical twins. It is clear, then, that the effects on ductility of the first three factors listed above are complex and interdependent.

A microstructure of large lamellar grains gives low ductility ², because the high yield stress of lamellar grains is highly anisotropic, with respect to the lamellar orientation. The strength is high when the lamellae are parallel or perpendicular to the loading axis, and low for intermediate orientations ¹²⁰. In the former situation shear must occur across lamellar boundaries, while in the latter case shear can occur along lamellae interfaces, which provides easier deformation. Thus, large strain discontinuities arise at boundaries between large lamellar grains depending upon the orientation of the lamellae of adjacent grains. Such a strain discontinuity would lead to crack initiation at the grain boundaries. If a single phase γ -TiAl grain exists between two lamellar grains, the strain discontinuity which would arise if they were neighbours disappears, since the γ -TiAl grain deforms by microcracking at room temperature, dynamic recrystallization and twinning at elevated temperatures (700°C) ¹²¹. This explains why the ductility of the two phase duplex structure is highest and depends on the ratio of the volume fraction of single phase grains to that of lamellar grains ¹¹⁶. The best balance of tensile strength and ductility is obtained with a duplex structure which contains a lamellar grain to γ grain ratio of $\approx 30\%$ ²¹. The effects on tensile properties of these microstructural variables are

summarized in Table IV ²⁴, in which three of the heat treatments correspond to the microstructures of Figure 8.

A positive temperature dependence of yield stress was first reported for TiAl single crystals ¹²², and has since been observed for several coarse grained ternary alloys ^{123,124}. The increasing yield stress with temperature was correlated to the tendency for superdislocations to dissociate into fault dipoles, rather than dissociating into unit dislocations and twins ¹²⁵. The fault dipoles result from cross-slip of the $1/6[112]$ type partials from (111) planes to (110) planes. On this latter plane the partial dislocation is pinned due to a high Peierls stress ^{126,127}, resulting in localized pinning of the superdislocation. Superdislocation dissociation is dependent on both aluminum content and heat treatment temperature. Increasing aluminum content increases the stacking fault energy, while both aluminum content and heat treatment temperature alter the amount of second phase α_2 present, which it is postulated affects the superdislocation reactions, due to the tendency of α_2 to absorb interstitial elements. The yield strength of fine grained structures does not exhibit a positive temperature dependence because of the Hall-Petch strengthening affect at low temperatures ⁴⁴.

2.4.2 Fracture Toughness

The best room temperature fracture toughness reported for a near γ -TiAl alloy is $32 \text{ MPa}\sqrt{\text{m}}$ ⁶ with binary Ti-48%Al exhibiting a toughness of only $15 \text{ MPa}\sqrt{\text{m}}$ ¹²⁸. It is noteworthy that ductility improvements, by the addition of alloying elements, do not result in corresponding fracture toughness improvements ^{129,130}, but rather fracture toughness depends on microstructure. The fracture toughness is very sensitive to strain rate, resulting in Charpy impact values of only 1 J obtained to date ¹³¹. Clearly the dynamic fracture toughness needs improvement before damage tolerant applications are possible.

The major microstructural feature affecting the fracture toughness is the volume fraction of lamellar structure present, which may be altered by heat treatment. The

transition from a structure dominated by equiaxed γ grains to one containing fully transformed lamellar structure (see Figure 8) results in almost doubling of the fracture toughness ⁶, see Table V. In the γ phase, fracture occurs by grain boundary decohesion and cleavage ^{6,37,130}, with the major mode of deformation being movement of $\frac{1}{2}[110]$ dislocations. Cleavage is primarily due to dislocation pile ups and covalent bonding causing high cohesive strength. Thus the γ phase has a high flow stress relative to cleavage stress and so fracture is easier than plasticity ¹³².

The lamellar structure resists fracture more effectively, since cracks can be arrested by lamellae perpendicular to the advancing crack, Figure 34 ¹³⁰. However, similarly to the tensile strength of lamellar regions, the fracture resistance of lamellar regions is highly anisotropic. As such, fracture proceeds by brittle failure through lamellar colonies oriented parallel to the crack front (Figure 34b). This is accompanied by mechanical twinning in the γ laths parallel to the crack ²⁵. If secondary lamellar colonies are present, these can act as 'bridging ligaments', thus arresting and forcing renucleation of the crack. The bridging ligaments of secondary lamellar colonies eventually fail by debonding, but do serve to increase the fracture toughness ²⁵. Grain boundaries between lamellar grains resist crack growth due to the ragged interlocking structure of the laths at grain boundaries ¹³¹.

In section 2.4.1 a duplex microstructure of $\alpha_2 + \gamma$ lath and equiaxed γ grains was shown to give the best combination of strength and ductility. As seen in Table II, such a microstructure has considerably better fracture toughness than single phase equiaxed γ . This improved toughness is due to ¹³³:

- a) the smaller grain size of the duplex structure,
- b) the γ laths in the lamellae having four variants of orientation (see section 2.1), providing more freedom for deformation,
- c) grain boundaries of the duplex structure being free of oxygen and nitrogen due to scavenging by the α_2 phase (see section 2.5.8).
- d) the occurrence of mechanical twinning in the region adjacent to the advancing crack ¹³⁴.

As the temperature is increased all microstructural conditions exhibit a more ductile failure, along with a propensity for separation along grain boundaries. These changes bring about increased toughness ¹³⁵. However, only at temperatures above the brittle to ductile transition do the mechanisms of cross twinning and dislocation climb operate effectively to suppress planar deformation and cleavage ¹³³. The grain boundary separation is caused by their low cohesive strength, which is due to impurities segregating to boundaries, because of low energy interstitial sites.

The grain boundary cohesive strength is also lowered in ordered structures because disruption of the lattice at grain boundaries is more severe than normal due to the complex superlattice structures. This weakens the grain boundary considerably compared to the grain interior ¹³⁶. This situation is exacerbated by the different charge densities surrounding the two constituent elements. Aluminum will tend to withdraw electrons from titanium because of its higher electronegativity. Consequently, in areas of severe disruption, like grain boundaries, the strength of potential Ti-Ti bonds across the boundary is lowered ¹³⁶. Clearly, this can be overcome by alloying with ternary elements that have low electron attractions and will diffuse to grain boundary locations. This possibility is discussed in section 2.5.

2.4.3 Creep Properties

When compared on a normalized density basis, The TiAl compound has stress-rupture properties superior to those of nickel base superalloys as shown in Figure 35 ¹³⁷. The creep deformation of TiAl produces a strain versus time curve, Figure 36 ¹³⁸, similar to that for other high temperature metals. These curves are characterized by an initial stage of decreasing strain rate with time, caused by the formation of a more creep resistant sub-structure. This is followed by a steady state or minimum creep strain rate, due to stabilization of the sub-structure by some recovery process ¹³⁹. Finally a period of increasing strain rate leads to failure. For gas turbine applications creep resistance is of prime concern, with many critical components designed to a maximum creep strain of

1% ¹³⁹. The nickel base superalloys have primary creep strains of <1% and very low secondary or steady state creep rates. Similar properties are required of titanium aluminides.

For compositions of Ti-51.5%Al and Ti-53.4%Al grain size affects creep rate minimally, over a wide range of stresses and temperatures ¹⁴⁰. In contrast, for Ti-50%Al the grain size exerts a strong effect on the creep strain rate ¹⁴¹. For 50%Al the relationship between minimum creep strain rate ($\dot{\epsilon}_m$) and grain size (d) at high stresses is $\dot{\epsilon} \propto d^{-1}$ and at low stresses is $\dot{\epsilon} \propto d^{-3}$ ¹⁴². Clearly, the effect of grain size on the creep properties is not solely due to grain boundary sliding, as this doesn't explain the compositional differences, however a low stress $\dot{\epsilon} \propto d^{-3}$ relationship is typical of grain boundary diffusional creep ¹⁴¹.

The dependence of the minimum creep rate of Ti₃Al ¹⁴⁴ and TiAl ¹⁴⁵ on temperature and stress obeys the power law creep equation:

$$\dot{\epsilon} = A \left(\frac{\sigma}{E} \right)^n \cdot e^{-Q/RT} \quad \dots (14) .$$

In this equation the activation energy Q is the slope of the minimum strain rate versus the inverse of temperature and the stress exponent n is the slope of the minimum strain rate versus stress curve. The constant A, is a structure dependent constant which reflects the influence of stacking fault energy ¹⁴⁶. For single phase TiAl the minimum strain rate as a function of stress or temperature exhibits a change in slope as shown in Figure 37 ¹⁴⁷. Therefore the activation energy or creep rate controlling factor changes from low to high strain rates ¹⁴⁸. Some important characteristics of this observation are:

1. the transition between the rate controlling factors occurs at roughly the same creep strain rate ($3 \text{ to } 5 \times 10^{-5} \text{ sec}^{-1}$) independent of temperature ¹⁴⁹,
2. the minimum creep strain rate is more dependent on stress at low stresses than at high stresses ¹⁴⁹, and
3. temperature dependence in the low stress region is larger than in the high stress region ¹⁴⁸.

A change in the value of the stress exponent, n , is also observed for two phase near γ -TiAl alloys, Figure 38¹⁵⁰. Note that single phase (Figure 37) and two phase (Figure 38) alloys display similar stress exponents at strain rates of $\approx 10^{-6}$ to 10^{-7} s⁻¹. The reported values for the creep activation energy varies from 300 to 410 kJ/mole^{145,148,150,151,153}, and depend on the applied stress. The changes in stress exponent and activation energy suggest a change in the controlling creep deformation mechanism. However, the activation energy for interdiffusion between pure Ti and Ti-47 to 54%Al is only 130 to 165 kJ/mole¹⁵⁴, which would seem to imply the rate controlling process is more complex than simple diffusion. However, it is inappropriate to compare the activation energy for creep with that for interdiffusion, since interdiffusion coefficients measure the fastest moving atomic species, whereas the creep deformation rate is controlled by the slowest moving atomic species¹⁰⁴. It has been suggested that pipe diffusion may be the rate controlling process, since this can double the stress exponent¹⁵⁰. Thus, the large change in stress exponent of Figure 38 (and corresponding increase in activation energy) is possibly explained by diffusional creep at low stresses with a change to dislocation creep at high stresses¹⁵⁰. Both these mechanisms are controlled by diffusion via dislocation pipes.

For single phase TiAl, deformation during primary stage creep (up to about 0.3 true strain) is characterized by the formation of twins and cross twinning¹⁴⁰. Increasing strain causes pronounced bending of the twin bands, suggesting dislocation glide in the matrix. The structure becomes gradually finer with grain partitioning imposed by deformation twinning and cross twinning. By 0.5 true strain, fine dynamically recrystallized grains are observed, which cover the entire area by ≈ 0.8 true strain. Dynamic recrystallization of two phase alloys occurs preferentially along grain boundaries during creep deformation^{155,156,157}. As the bulk strength increases, dynamic recrystallization becomes more difficult and creep resistance increases¹⁵⁶. Apparently, the formation of a fine dynamically recrystallized structure allows primary creep deformation to occur again and the creep rate is increased¹⁵⁸. Dynamic recrystallization along grain boundaries indicates that grain boundary sliding also could be contributing to creep deformation, since the extent of recrystallization is a function of the local plastic strain. It appears, then, that

creep deformation in near γ -TiAl alloys is controlled to some extent by dynamic recrystallization.

For binary Ti-48%Al the creep resistance is dependent on the microstructure, with a 'duplex' structure exhibiting significantly higher creep rates than a fully lamellar structure ¹⁵⁹. This result may be due to the larger grain size often observed for the fully lamellar structure. However, a characteristic of the lamellar structure is interlocking grain boundaries, which may inhibit grain boundary sliding during creep. Thus, the relative effect of the grain size and the lamellar or duplex structure on creep properties is still ambiguous. Note that the lamellar structure is superior in creep and fracture toughness (Table V) compared to the duplex structure, but results in the lowest tensile ductility (Table IV).

Creep fracture of two phase compositions is very sensitive to oxidation ¹³⁸, with the time to rupture (at 900°C) in vacuum increased by more than a factor of ten over the rupture time in air. Creep fracture in air and vacuum is mainly intergranular, with deformation occurring by $\frac{1}{2}[110]$ dislocations in the γ grains of γ platelets of the lamellar structure only. This is in agreement with the deformation behaviour reported in section 2.2.

2.4.4 Fatigue Properties

Ambient and elevated temperature fatigue crack growth rates for Ti-48%Al in air have been determined recently ^{128,160}. The results obtained for precracked single edge notched specimens are summarized in Figure 39 ¹⁶¹. Note that the fatigue crack growth rates at 700°C are about an order of magnitude less than those obtained at ambient temperature and a higher threshold stress intensity is obtained. The improved elevated temperature fatigue crack growth rate was attributed to oxide induced closure ¹⁶⁰. No evidence of sub-structure development was evident at the 700°C test temperature. This was taken as an indication that reversed plasticity is not responsible for fatigue crack

growth in this compound. However, twinning deformation did occur during fatigue loading at 700°C, but the relationship between twinning and fatigue crack growth was not established. It was also noted ^{160,161}, that fatigue crack growth occurs by cleavage across γ grains and by crystallographic fracture across lamellar grains, producing a more torturous crack path, which is consistent with the failure morphology described in section 2.4.2.

2.4.5 Mechanical Properties of HIP'ED γ -TiAl

Enhanced bonding strength between prior particles is achieved with higher HIP temperature, as confirmed by the increasing ultimate strength shown in Figure 40 ⁶¹. This corresponds to more complete densification at the higher HIP temperatures and fracture by cleavage. In contrast, at lower HIP temperatures (900°C) the fracture surface exhibits regions of cleavage and dendritic structure retained from the powder solidification microstructure.

The compressive yield strength of HIP consolidated Ti-50.9%Al is shown in Figure 41 ⁶⁴. The compressive yield strength is constant at ≈ 390 MPa up to a temperature of $\approx 800^\circ\text{C}$ (1073K) and then decreases rapidly. Note that the 1% and 2% offset stress exhibits similar behaviour to the yield stress, indicating that the work hardening rate does not change in the temperature range from ambient to $\approx 700^\circ\text{C}$ (973K) and then decreases above this temperature. The compressive ductility of the HIP'ed powder was $>26\%$ at room temperature, but the bending fracture strain was $<0.5\%$. This suggests that ductility can be increased if cracking and cavity formation can be suppressed.

Heat treatment or hot working of HIP'ed TiAl improves the ductility and reduces the tendency for prior particle boundary failure at elevated temperatures ⁶⁴. For Ti-48%Al-1%V a suggested post-HIP heat treatment is 1260°C/4 hr ($\gamma+\alpha$ phase field) air cool, plus 1093°C/8 hr ($\gamma+\alpha_2$ field) air cool. This heat treatment results in some grain growth and a slightly higher volume fraction of intergranular α_2 . The tensile properties resulting from

this post-HIP heat treatment are listed in Table VI ⁷³. Note the low ductility achieved until the brittle-ductile transition temperature ($\approx 700^\circ$) is reached. This behaviour is common to most γ -TiAl compositions whether produced by PM or ingot processing techniques. The corresponding stress-rupture properties are plotted in Figure 42 ^{73,138,156,162} and compared to similar properties for wrought material of the same composition.

2.4.6 Mechanical Properties - Summary

From the preceding discussion it is clear that the mechanical properties of near γ -TiAl alloys are very dependent on microstructure. Furthermore, there are trade-offs among the mechanical properties for each microstructure, which are summarized in Table VII ⁴⁴. It is necessary to realize when examining Table VII that the fully transformed lamellar structure generally has a larger grain size, due to fast growth during heat treatment in the single phase α phase field. Thus, it is not always clear if the mechanical property differences are due to grain size or microstructural effects. Indeed, the metallurgical mechanism controlling many of the mechanical properties is still uncertain. A useful combination of properties may be attained if a fine grained fully lamellar microstructure could be developed.

2.5 TERNARY ADDITIONS

The applications envisaged for titanium aluminides require a demanding combination of properties. To better meet these requirements a considerable amount of research has dealt with the effects of ternary additions on the processing and properties of TiAl. One major reason to add a third element is to improve ductility. Titanium and aluminum have a covalent bonding character due to the strong interaction between the aluminum 'p' and titanium 'd' electrons. This interaction is in large part responsible for the cohesive strength of TiAl ²⁶. As discussed previously, one reason why the TiAl intermetallics fail is because their cohesive bond strength exceeds the transgranular cleavage strength. Thus it has been suggested that ductility is in some manner related to

the nature of this chemical bond, and lowering of the p-d interaction of the bonds should improve the ductility ²⁶. One way to achieve this, is the addition of transition elements which substitute for aluminum in the superlattice. This should strengthen the d-d bonding component and lower the anisotropy of the bonds. Clearly, however, many other properties of titanium aluminide intermetallics are influenced by the addition of ternary elements, for example, unit lattice volume, grain size, phase stability, precipitation of second phases, etc. These other factors may mask the effect due to changes in the electronic bond character. However, by considering the bond character some potentially beneficial ternary elements can be identified, for example V, Cr and Mn. Bearing in mind the preceding discussion, the properties of the various elements will be considered individually.

2.5.1 Chromium

The addition of up to 4% chromium to TiAl decreases the unit cell volume, Figure 43 ^{19,46}, as do many other ternary elements. The significant increase in the yield and fracture stress caused by the chromium addition is illustrated in Figure 44 ¹⁶³. The effect of chromium on the fracture strain (ductility) depends on whether it is substituted for aluminum or titanium. As seen in Figure 44 substitution of up to 3% chromium for aluminum increases fracture strain, while substitution of 3% chromium for titanium causes a major drop in fracture strain.

The effects of chromium on mechanical properties can be correlated to microstructural changes. When chromium is substituted for aluminum the grain size decreases and the volume fraction of lamellar phase increases (even though the percentage of chromium in the γ and α phases is the same) ¹⁶³. As discussed in section 2.4.1 both these effects are beneficial for ductility. With the increase in ductility a larger area fraction of intergranular fracture is observed. The deformation of chromium containing TiAl differs from the binary compound in that it has a much higher propensity for

twinning ¹⁶³, due to alignment of $1/6[121]$ partial dislocations on neighbouring planes ¹⁶⁴ and a reduction in stacking fault energy caused by modification of the Ti-Al bond ¹¹⁹.

In rapidly solidified TiAl containing chromium and 0.5% tantalum the precipitation of fine TiCr_2 has been reported ¹⁶⁵. In a 3% chromium composition after annealing at 900°C for 4 hours, the precipitates were $<1\ \mu\text{m}$ and covered an area fraction of 2%. With 5% chromium the TiCr_2 precipitates preferentially at grain boundaries and prevents grain coarsening during annealing which occurred in the non-chromium containing composition. Due to the finer microstructure and higher bulk diffusivity, TiCr_2 particles lower the flow stress at elevated temperatures. A small amount of carbon (about 1350 ppm) is required to stabilize the TiCr_2 precipitates. The possible beneficial effect of such precipitates on creep properties does not appear to have been investigated.

The addition of chromium promotes the formation of body centred cubic β Ti_2AlCr ¹¹⁹. This β phase may form preferentially within the α_2 laths with the orientation relationship $(0001)_{\alpha_2} \parallel \{\bar{1}11\}_{\gamma} \parallel \{110\}_{\beta}$ and $\langle 11\bar{2}0 \rangle_{\alpha_2} \parallel \langle 110 \rangle_{\gamma} \parallel \langle 1\bar{1}1 \rangle_{\beta}$ ¹⁶⁶. This formation of β within the α_2 laths causes the lamellar structure to become unstable and readily break-up into irregularly shaped particles of α and β in a γ matrix ¹⁶⁷. The β acts as a hinderence to slip in the γ phase and refines the grain size. Both these factors enhance ductility ¹⁶⁷. In plasma arc melted and homogenized $\text{TiAl}+3\%\text{Cr}$, isothermal forging at 800°C and $7.5 \times 10^{-4}\ \text{sec}^{-1}$ caused the formation of chromium enriched β phase at grain boundaries ¹⁶⁸. As outlined in section 2.3.3.2, superplastic tensile elongations of $>450\%$ were attained for this alloy when tested at 1200°C. It was concluded that β phase formation at grain boundaries is necessary for improving ductility of γ -TiAl alloys.

2.5.2 Manganese

Manganese is one of the few elements that occupies mainly the aluminum sites in the TiAl superlattice ⁵⁵. The effect of this is to reduce the covalent bond character between the aluminum and titanium and soften the TiAl phase.

The second major effect of manganese additions is to greatly increase twinning deformation. The extra deformation mode of twinning improves ductility and strength as evidenced by Figure 45 ¹⁶⁹. Furthermore the addition of 2% manganese thermally stabilizes the $(11\bar{1})[112]$ twins which still exist after annealing 24 hours at 1000°C. The twin stability is caused by manganese segregating to twin boundaries and pinning the partial dislocations. Manganese segregated at the twin boundaries acts as nucleation sites for superdislocations ¹⁶⁹. The mobility of the superdislocations is increased since manganese eliminates most of the pinning effect of the $1/6\langle 112 \rangle$ partial dislocations at room temperature ¹⁷⁰, resulting in improved ductility.

2.5.3 Vanadium

The addition of 1% vanadium to duplex TiAl compounds produces an improved balance of strength and ductility (507 MPa ultimate strength and 2.3% elongation at room temperature for Ti-48%Al-1%V ²). The increase in ductility is only apparent when vanadium is substituted for aluminum ⁵⁵. When substituted for titanium the yield strength is increased substantially, but with no effect on ductility ¹⁷¹.

The vanadium will occupy both the aluminum and titanium sites in the superlattice in duplex alloys, but only titanium sites in single phase alloys and reduces the unit cell volume ¹⁷², see Figure 43. However, the tetragonality remains nearly constant. The improved ductility is the result of the formation of profuse cross twinning (across grain boundaries) of the $(11\bar{1})[112]$ type ¹⁷¹, weakening of the Ti-Al covalency and promotion of twin deformation ¹⁷².

A positive temperature dependence of the yield stress exists for TiAl+10%V ¹⁷³. This was attributed to cross-slip of $1/2[110]$ dislocations, which unlike the case for fcc metals, results in a reduction of the dislocation mobility and an increase in dislocation density. Additionally, pinning of $1/2[110]$ dislocations by superdislocations and mutual

pinning of superdislocations were considered significant factors. A sharp drop in yield above 800°C was thought to be due to a decreasing Peierls stress.

2.5.4 Tungsten

Adding ternary tungsten to TiAl results in several beneficial changes to mechanical properties. Foremost of these changes is increased tensile strength with a small drop in room temperature elongation. Table VIII ^{117,152,162} compares the tensile properties of binary TiAl and Ti-49%Al-2%W produced from PREP powder and consolidated by extrusion. The increased tensile strength is attributed to the additive effects of a finer grain size, reduced slip length and a small amount of solid solution strengthening. Note that the ductility of the W ternary alloy does not exhibit a pronounced ductile-brittle transition at $\approx 700^\circ\text{C}$ as observed in many near γ -TiAl alloys. It is postulated ¹⁵² that this is due to the saturation of the matrix with W, inhibiting dislocation movement at elevated temperatures. In contrast, small ingots of Ti-48.5%Al-0.5%W exhibited 31% elongation at 800°C ¹⁷⁴. Tungsten reduces the unit cell volume and tetragonality of TiAl ¹⁶¹, and therefore would be expected to improve ductility since the $\langle 110 \rangle$ slip directions become more equivalent. However note from the data of Table VIII, that this ductility improvement fails to occur.

The steady state creep strain rate is lowered by adding tungsten to TiAl ^{145,152}, see Figure 46. There are several reasons for the improved creep resistance ^{117,130,145,152}:

- i) Nucleation of tungsten rich precipitates on the grain boundaries inhibits grain boundary sliding, lowering the minimum creep rate even though a finer grain size exists.
- ii) A fine dispersion of tungsten particles within the grains retards the passage of dislocations, which must bypass these particles by either Orowan bowing or climb processes.
- iii) The presence of the low diffusivity tungsten solute impedes diffusion assisted dislocation movement.

This last mechanism is the most effective at improving the creep resistance, as evidenced by the results of Table IX^{152,162}. In the as extruded condition the tungsten is held in super saturated solid solution, but forms tungsten rich β body centred cubic particles during heat treatment. The precipitation kinetics of the tungsten rich particles are sufficiently slow that elevated temperature exposure during creep strain does not cause nucleation and growth of this phase even on dislocations¹⁵². As seen in Table IX, the time to 1% creep strain is about double in the as extruded condition compared to the heat treated states. The lower minimum (steady state) creep strain for the as extruded material, Figure 43, is also consistent with the results of Table IX.

The creep activation energy of as extruded Ti-48%Al-2%W is ≈ 400 kJ/mole¹⁵², in comparison to 370 kJ/mole for the same composition in the heat treated condition and 300 kJ/mole for Ti-50%Al¹⁴⁵. These differences in activation energies are consistent with the trends of Figure 46 and Table IX, but are not sufficient to explain the large changes in minimum creep rate (Figure 46) or time to 1% strain (Table IX). The increased creep resistance has been rationalized by suggesting that tungsten solid solution strengthening has a linear impact (affects only the constant A in equation 11 of section 2.4.3) on the creep strain rate without affecting the stress or temperature sensitivity¹⁵². It is speculated that the tungsten addition lowers the stacking fault energy¹⁴⁶, especially when present in solid solution, thus increasing the activation energy of the extruded condition.

The oxidation rate of TiAl is significantly reduced by the addition of between 2 and 4% tungsten^{175,176} as seen in Figure 47. The reason for the improved oxidation resistance is not clearly understood, but it appears that the addition of tungsten (or niobium or Nb+Cr) facilitates the growth of protective alumina scales even at low aluminum contents¹⁷⁷.

2.5.5 Niobium

In TiAl additions of up to 4% niobium have the effect of decreasing the unit cell volume as seen in Figure 43. However, in contrast to other elements that reduce unit cell volume, niobium additions decrease the fracture strain substantially. TiAl containing 3% niobium exhibits only half the fracture strain of binary TiAl, see Figure 48¹⁶⁸. The yield and fracture stress increase sharply with niobium content, due to the large solubility of niobium in TiAl which would be expected to produce effective solid solution strengthening¹⁷⁸. Interestingly, with these changes in tensile properties, there is no corresponding affect on fatigue life. Fatigue S-N curves for TiAl with or without niobium are similar at room temperature and 900°C¹⁷⁹.

Like vanadium additions, niobium can cause increasing yield stress with temperatures between 400 and 900°C¹⁷⁸. The yield stress of TiAl with 5% niobium is compared to binary TiAl in Figure 49. The increasing yield strength was noted to be coincidental with the strengthening mechanism outlined in the earlier section on vanadium. Niobium also lowers the temperature at which twinning becomes an important deformation mode, thus lowering the ductile-brittle transition temperature¹⁷⁸.

2.5.6 Carbon

A small amount of carbon (up to 0.2%) has a profound affect on creep rates of TiAl. An addition of 0.1% carbon to Ti-48%Al-1%V improves creep resistance by a factor of six¹⁸⁵, but with a drop in ductility. The addition of carbon to Ti-55%Al results in the precipitation of Ti_3AlC carbides¹⁸⁹, homogeneously in the γ matrix and heterogeneously along dislocations and grain boundaries. Grain boundaries are strengthened by carbon, since carbon occupies sites that may otherwise be embrittled by hydrogen or oxygen. This grain boundary effect may account for the improved creep resistance. It may also be that the improvement in creep resistance is analogous to that resulting from the presence of trace amounts of nitrogen, which is discussed in section 2.6.8. That is, the

precipitation of carbides during stage I creep deformation hinders the movement of dislocations.

2.5.7 Boron

Boron has been added to TiAl by utilizing the XDTM process that was outlined in section 2.3.2. In this process the boron is added as titanium diboride particles and a microstructure containing TiB₂ particles and interstitial boron results ⁽⁶⁾. Interstitial boron occupies the octahedral sites and reduces the lattice tetragonality. As the amount of interstitial boron increases the structure becomes closer to face centred cubic and the formation of three sets of <110> dislocations is favoured. This improves the ability of the microstructure to accommodate plastic deformation. Three sets of dislocations have been observed in Ti-46%Al+7.5vol%TiB₂ ⁽⁶⁾.

The effect on the microstructure of various amounts of TiB₂ is shown in Figure 50 ⁽⁸¹⁾. It is evident that the initial addition of TiB₂ causes a large drop in the colony size, with further amounts of TiB₂ causing only small further decreases in colony size. It can be postulated that only a small number of TiB₂ particles are required to increase substantially the nucleation rate of solidification. TiB₂ melts at 3225°C and so would act as solid particles suspended in the melt. The TiB₂ particles resulting from this process are about 1µm ⁽⁸²⁾, and are not altered by thermal mechanical processing. TiAl that is XDTM reinforced by TiB₂ has significantly improved strength at all temperatures compared to binary TiAl ⁽⁸³⁾. Tensile properties of Ti-47%Al+6vol%TiB₂ are listed in Table X ⁽⁸⁴⁾. Note in Table III that despite the reduced colony size tensile ductility is still <1%. The decreasing hardness, yield strength and increasing plastic elongation is associated with an increasing volume fraction of primary equiaxed TiAl grains.

In XDTM TiAl reinforced with TiB₂ an interesting feature is the formation of Ti₃Al at the boundary of twinned regions during deformation ⁽⁸⁵⁾. This characteristic does not appear to be related to the presence of TiB₂. The presence of Ti₃Al at twin boundaries

eliminates the requirement for symmetry between twinned regions and all $\langle 112 \rangle \{111\}$ type twins can occur ¹⁸⁶. The formation of Ti_3Al at twin boundaries has been explained in the manner of Figure 7. That is, stacking faults of TiAl have the same structure as Ti_3Al and so could act as nucleation sites for Ti_3Al . Similarly, a Ti_3Al stacking fault could act as a nucleation site for the twinned TiAl . Repetition of this sequence results in a $\text{TiAl}/\text{Ti}_3\text{Al}$ lamellar structure.

It was observed that further deformation during hot rolling causes recrystallization to occur more readily at twin and grain boundaries at which $\text{TiAl}/\text{Ti}_3\text{Al}$ lamellae had previously formed ¹⁸⁶. More interesting is that for both $\text{Ti-46\%Al}$ and XD^{TM} produced $\text{Ti-47\%Al}$ with TiB_2 , creep deformation, at 69 to 138 MPa and 760°C , caused successive cycles of lamellar structure formation and recrystallization ¹⁸⁷. These microstructural changes during creep would appear to be independent of the presence of boron in solid solution or TiB_2 particles and were concluded to be the predominant creep mechanism.

2.5.8 Trace Elements

The presence of trace amounts of the impurity elements oxygen and nitrogen has a considerable effect on microstructure and mechanical properties. Stoichiometric TiAl produced from super high purity material exhibits a single phase equiaxed structure compared to a fine lamellar structure for ordinary purity TiAl ¹⁸⁸. Furthermore, the room temperature fracture strain of the high purity TiAl was $>3\%$, as compared to 1% for ordinary purity TiAl . Thus, purification causes microstructural changes which result in enhanced ductility. It is likely, therefore, that trace amounts of oxygen and nitrogen considerably affect the location of the phase diagram boundaries. Both oxygen and nitrogen have a low solubility in the γ phase and so in two phase alloys are interstitial in the α_2 phase, leaving the γ phase and grain boundaries free of both elements ^{46,189}. In two phase TiAl compounds oxygen and nitrogen stabilize the α_2 phase. In single phase TiAl compositions these interstitials will be supersaturated in the γ matrix and segregate to

grain boundaries and defects ⁴⁶. Such segregation affects deformation modes by altering dislocation movement and lowering ductility.

In single phase TiAl compounds, small amounts of nitrogen (as little as 40 ppm) cause the formation of Ti_2AlN nitrides during heat treatment between 1000 and 1100°C ¹⁹⁰. Furthermore, during creep deformation between 750 and 950°C, Ti_2AlN precipitated on the forming mobile dislocations, subsequently rendering these dislocations immobile and thus lowering the stage I creep rate, as shown in Figure 51 ¹⁹¹. Once the precipitation of Ti_2AlN (the kinetics of which are very slow) has consumed all the available nitrogen, additional dislocations formed by the creep deformation may move unhindered by Ti_2Al precipitates and the creep rate increases to the stage II steady state rate.

This mechanism of reducing the creep rate by nitride precipitation may be achievable with other elements. For example, it is not clear if the formation of carbides causes the same affect on creep rates as the nitrides. It is possible that if this affect were possible with other ternary elements, the creep rate could be reduced while also obtaining other beneficial affects.

3. MATERIALS AND EXPERIMENTAL PROCEDURE

3.1 POWDER COMPOSITION

This thesis work represents the first experimental work on near γ -TiAl undertaken at Carleton University or the National Research Council Canada. As such, an important first task of this thesis investigation was the selection of a suitable intermetallic composition for investigation. Due to the high cost of procuring powder, the information contained in the previous chapter was carefully considered, such that one composition that would fulfil two main criteria could be identified. Firstly, the chosen alloy should be capable of generating mechanical properties with attributes beneficial for gas turbine applications. Secondly, it was considered desirable that the selected composition present opportunities for investigation of the factors controlling various metallurgical processes of near γ -TiAl.

Based on the selection rational of the previous paragraph, a nominal composition of 50%Ti-48%Al-2%W was selected for this thesis investigation. This composition should possess a number of beneficial characteristics, including:

1. a fine grained microstructure due to below stoichiometric aluminium content,
2. increased tensile strength caused by the fine grained structure and tungsten solid solution strengthening,
3. improved creep resistance, study of which may enhance the understanding of the metallurgical mechanisms controlling creep of near γ -TiAl,
4. significantly improved oxidation resistance, and

5. the presence of tungsten rich β phase may enhance superplasticity (as is the case for TiAl+Cr, see section 2.3.3.2), thus aiding the understanding of this phenomenon.

These characteristics are reviewed in the previous chapter. It is clear that the investigation of all the above aspects of TiAl+W compositions is beyond the scope of this thesis. However, it is hoped that the availability of this powder will allow further study of this ternary system.

3.2 POWDER PROCUREMENT

The powder was procured from Crucible Compaction Metals Inc. and was produced by the 'titanium gas atomization' (TGA) process outlined in section 2.3.2.2. The chemical analysis of the Ti-48%Al-2%W powder is listed in Table XI¹⁹², along with the analysis for a small quantity of binary Ti-54%Al¹⁹³ powder which was also obtained from Crucible Research, to provide a basis for comparison. Ideally, a binary Ti-48%Al composition would be preferred for comparison, but was not available. Note the very low impurity (C, N, O and H) levels. Figure 52 compares the powder size distribution of the two powder samples with the distribution of Figure 17, which is apparently typical for γ -TiAl TGA powder. Two differences observed are; a) no Ti-54%Al powder was $<105\text{ }\mu\text{m}$ size and b) the remaining Ti-54%Al powder is coarser than the typical TGA γ -TiAl size distribution. The former difference is due to a prior screening of fine powder from the Ti-54%Al sample. It is not clear if this screening process can account for the latter difference. The Ti-48%Al-2%W powder size distribution is not significantly different from the typical distribution. The Ti-48%Al-2%W was received in sealed cans, pressurized with clean dry air. The Ti-54%Al powder had received a considerable degree of handling and exposure to atmospheric air. The flow rate of the powder is 32 and 36 seconds for the ternary and binary compositions respectively.

3.3 METALLOGRAPHIC PREPARATION

Metallographic examination was undertaken using optical and SEM techniques. Sections for optical microscopy were prepared by polishing and etched by swabbing (5-8 sec) with Kroll's reagent*. Sections for SEM examination were in the as-polished condition unless indicated otherwise.

The preparation of metal for examination in the transmission electron microscope (TEM) involved thinning samples to a thickness of ≈ 100 nm. This was accomplished by cutting discs of 2.9 to 3 mm diameter and 0.25 mm thickness on a diamond wheel. These discs were thinned using emery cloth to ≈ 65 μ m and jet polished in an electrolyte of 5% perchloric acid in methanol until perforation occurred. In this manner TEM analysis of the metal adjacent to the perforation was possible. However, the jet polishing conditions had to be closely controlled to:

- electrolyte temperature -38°C to -42°C ,
- applied potential 25 to 30 V DC,
- current 125 mA,
- Struers Tenupol 3 electrolyte flow rate of 5, and
- Struers Tenupol 3 photosensitivity of 3.

3.4 TEM DISLOCATION IDENTIFICATION

The dislocations active during deformation of near γ -TiAl were reviewed in section 2.2. One objective of this thesis was to determine the Burgers vectors of the dislocations contributing to deformation. This was achieved by diffraction contrast imaging in the TEM. This section briefly outlines the principles and procedure for this technique.

* Kroll's Reagent: 100 ml H_2O , 5 ml HNO_3 , 2 ml HF.

From electron microscopy diffraction contrast theory ¹⁹⁴ it can be shown that dislocations can only be imaged in the TEM when;

$$\mathbf{g} \cdot \mathbf{b} \neq 0 \quad \dots (15)$$

where $\mathbf{b}$ is the dislocation Burgers vector and $\mathbf{g}$ is the vector normal to the crystallographic plane causing diffraction of the incident electron beam. It should be evident that for a random orientation between the incident electron beam and a particular grain, many planes will cause diffraction (that is, satisfy the Bragg equation). Thus, essentially all dislocations will be imaged. However, if for an orientation of incident electrons and the imaged grain $\mathbf{g} \cdot \mathbf{b}_1 = 0$, then the image of dislocations with Burgers vector $\mathbf{b}_1$ will be extinguished.

For the face centred tetragonal TiAl structure, the dislocations of interest are $\frac{1}{2}\langle 110 \rangle$, $\frac{1}{2}\langle 112 \rangle$ and $\langle 101 \rangle$ type dislocations. For these dislocations the vector product of the Burgers vector with the $1\bar{1}1$, $\bar{1}11$, and 002 $\mathbf{g}$ -vectors are listed in Table XII. Note that only the superdislocations $\frac{1}{2}\langle 112 \rangle$ and $\langle 101 \rangle$ are imaged with a $\mathbf{g}$ -vector of 002 . Furthermore, dislocations which are imaged clearly with all three $\mathbf{g}$ -vectors are of the $\frac{1}{2}\langle 112 \rangle$ type. Thus, to positively identify dislocations the $\mathbf{g}$ -vectors of the lattice planes causing diffraction must be determined.

The lattice planes corresponding to the three $\mathbf{g}$ -vectors of interest all cause diffraction when the incident electron beam is parallel to the $[110]$ direction. Hence, the sample is tilted with respect to the incident beam until the selected area diffraction (SAD) pattern for the grain of interest corresponds to that for the $[110]$ direction. This procedure is simplified by realizing that when strong diffraction occurs by low index planes, the bright field image appears dark. Appropriate further tilting of the sample in two orthogonal directions, around the $[110]$ incident beam direction, will result in strong diffraction from only one lattice plane. The $\mathbf{g}$ -vector of the strongly diffracting plane is determined by examination of the SAD pattern in which the corresponding diffraction spot (aside from the transmitted beam spot) will appear prominent. This is commonly

referred to as the *two-beam* operating condition. By noting which dislocations images are extinguished for the two-beam condition corresponding to each g-vector, the dislocation Burgers vector can be determined from Table XII. Note that the $\frac{1}{2}[110]$ dislocations are never imaged using the two-beam conditions of Table XII. However, $\frac{1}{2}\langle 110 \rangle$ ordinary dislocations are most prominent in TiAl. Thus the lack of imaging $\frac{1}{2}[110]$ dislocations does not represent a problem, since the related $\frac{1}{2}[1\bar{1}0]$ dislocation will be imaged.

3.5 VACUUM CANNING OF POWDERS

In order to ensure successful HIP'ing of powders a standardized procedure for hermetically sealing powder was followed. The canning material used for all samples was 304 stainless steel. Typically one end of a length of stainless steel tubing would be crimped and sealed by tungsten arc welding. The tube was then degreased with petroleum ether. The Ti-48%Al-2%W samples of powder were loaded into the cans within a 'glove box' containing an argon atmosphere, which avoided contamination with foreign matter and exposure to atmospheric air at all times. The Ti-54%Al powder was cleaned in petroleum ether and then loaded into the cans since this material had already been exposed to ambient air for a considerable period.

The open end of the can was packed with glass wool to prevent powder being drawn into the vacuum system which is shown in Figure 53. This vacuum system consists of a roughing pump and the three stage diffusion pump shown in section in Figure 53. The pre-filled cans were evacuated to $<5 \times 10^{-5}$ mbar at room temperature and then heated to $\approx 300^\circ\text{C}$ and evacuated until a vacuum of $<5 \times 10^{-5}$ mbar stabilized. Vacuums as low as 2×10^{-7} mbar could be achieved.

The powder containing can was allowed to cool to room temperature and the filling tube hydraulically crimped and isolated from the vacuum system, while maintaining the crimping pressure. The filling tube was then tungsten arc welded between the crimping tool and glass wool, then the crimping pressure released.

3.6 HOT ISOSTATIC PRESSING OF POWDER

Samples of vacuum encapsulated powder (prepared in accordance with the procedure of the previous section) were HIP'ed in an Autoclave Engineers press. This unit is illustrated in Figure 54 and has a maximum operating temperature and pressure of 2000°C and 207 MPa. The specimen chamber dimensions are 240 mm by 120 mm. The usual operating procedure is to load the vacuum encapsulated powder within the press and purge with high purity argon three times to ensure that all atmospheric air is removed. The specimen chamber is then pressurized with argon to about 55 MPa before heating commences. The remaining pressure required is generated by the heating cycle, which takes about 1½ hours to heat from room temperature to the HIP temperature (usually 1000 to 1200°C for titanium aluminides). Cooling can be controlled to any rate slower than the power off furnace cool (about 750°C/hr). A typical HIP cycle temperature and pressure profile is shown in Figure 55.

3.7 TENSILE TESTS

The geometry and specifications of the tensile test specimens are illustrated in Figure 56. This sample configuration was selected so that a minimal amount of material was required. Also, the tooling required for elevated temperature tensile tests existed for this sample geometry.

Machining HIP'ed TiAl into the tensile sample geometry of Figure 56 involved considerable difficulty. Turning of the material on either a conventional engine lathe or numerically controlled lathe, at various cutting speeds and depths, produced unacceptable surfaces. Indeed, turning threads on this material was near impossible since the thread crests consistently broke. As a consequence of these problems machining of the threads and all finished surfaces was done by grinding followed by polishing. The details regarding these finishing operations are given in Appendix B ¹⁹⁵ and resulted in the excellent quality test specimen shown in Figure 56.

Tensile testing was performed using a computer controlled servo-hydraulic test system. For all tensile tests, strain measurements were obtained using an extensometer with quartz extension arms, as illustrated in Figure 57. In this manner the extensometer was located outside the furnace heating zone. For room temperature tests the loading rate was controlled to 100 MPa/min (initial strain rate of $1.5 \times 10^{-3} \text{ sec}^{-1}$). Selection of this slow loading rate avoided any sudden loads being applied to the tensile specimen when the test is started. This is important given that the room temperature dynamic toughness of TiAl is low. Since the room temperature strain rate sensitivity of metals is very low, the effect of this slow loading rate on tensile properties should be negligible. Elevated temperature tests were conducted at a constant stroke rate of 0.0025 mm/sec, corresponding to an initial strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$. Elevated temperatures were generated by the electric 'tunnel' furnace shown in Figure 57. Thermocouples attached to the sample at the middle of the gauge length and at the extensometer grooves controlled the furnace temperature to within 2°C of the set-point.

3.8 CONSTANT STRAIN RATE ISOTHERMAL COMPRESSION TESTS

The specimen geometry used for isothermal compression tests is shown in Figure 58. The specimen has a height to diameter ratio of ≈ 1.4 , which serves to minimize the frictional forces between the specimen and dies, while also minimizing buckling during compression. The concentric rings machined in the specimen ends are to help maintain a lubricant film between the die and specimen during compression. For the current tests, the lubricant consisted of a mixture of boron nitride powder and a boro-silicate glass frit. Using this lubricant for the compression of superalloy IN 713LC yielded a friction coefficient of <0.04 ¹⁹⁶.

The test rig used for the isothermal compression tests is shown in Figure 59. The design and operation of the apparatus has been described elsewhere ^{197,198}. The apparatus consists of two water cooled compression columns made from the heat resisting alloys specified in Figure 60. The test rig is capable of applying loads up to 100 kN at

temperatures up to 1200°C. The induction coil of Figure 60 heats the susceptor, from which radiation heats the specimen and dies. The temperature is controlled by two thermocouples embedded in the upper and lower compression tooling. Before heating commences, a vacuum is drawn in the test chamber, which is then flushed with argon several times. A slightly pressurized argon atmosphere is maintained during heating and compression.

The susceptor was fabricated from type 310 stainless steel or TZM (Mo-0.5%Ti-0.1%Zr-0.02%W). The latter alloy was selected because of its high melting point (2830°C). Unfortunately, TZM has very low magnetic susceptibility. Consequently, with the induction heater available sample temperatures >1125°C were not attainable with the TZM susceptor. For this reason, the 310 stainless steel susceptor was used for 1200°C tests. To prevent localized melting of the stainless steel susceptor ($T_m=1425^\circ\text{C}$) the induction power - and thus sample heating rate - was reduced at temperatures above $\approx 1000^\circ\text{C}$.

The load is applied to the specimen by a servo-hydraulic universal test frame. To maintain a constant strain rate, the die velocity must be proportional to the specimen instantaneous height. This requires that the die velocity decays exponentially with time. Such control is achieved with an analog function generator that sends an exponentially decaying signal to the input of the servo-hydraulic test frame. The function generator is described in detail elsewhere ¹⁹⁹. The device consists of a resistive-capacitive voltage decay circuit with an adjustable time constant. In this manner the true strain rate can be controlled to within $\pm 2\%$ ¹⁹⁹. This accuracy was confirmed by comparing the time required to reach various true strains with the calibration graph of Figure 61.

The specimen height is measured continuously by a linear variable differential transformer (LVDT), equipped with high temperature probes, that monitors the distance between the upper and lower dies. The LVDT output signal is fed back to the analog

function generator for strain rate control purposes. The control and instrumentation layout are shown schematically in Figure 62.

During compression the load applied (from the test frame load cell) and the specimen instantaneous height (from the LVDT) is recorded. This data provides true strain according to:

$$\epsilon = \ln \frac{h_o}{h} \quad \dots\dots (16) ,$$

where h_o is the specimen initial height,

h is the specimen instantaneous height.

Similarly, the true stress is calculated from:

$$\sigma_t = \frac{F \cdot h}{A_o \cdot h_o} \quad \dots\dots (17) ,$$

where F is the applied load,

A_o is the specimen initial cross-sectional area.

Note that in this formulation of true stress it is assumed that the specimen volume remains constant.

Directly after compression to the desired total strain, the sample is cooled as quickly as possible, to 'freeze in' the deformation microstructure. When possible, this was achieved by quenching the sample into water within ~10 seconds of completing the compression. However, the quenching mechanism proved to be very sensitive to the set up of each test. Consequently, not all samples could be successfully quenched. In such cases, the furnace chamber was flushed with argon. This resulted in the cooling rate plotted in Figure 63. Over the temperature range from 1200°C to 800°C this corresponds to a cooling rate for the platens of 0.5°C/second. The cooling rate of the test sample would be somewhat higher, due to its lower thermal inertia.

4. POWDER MICROSTRUCTURE

The microstructures of the titanium gas atomized powder of both compositions were examined in the 'as-received' state from Crucible Compaction Metals Inc. An understanding of the as-received microstructures is necessary in order to decode the microstructural evolution occurring during powder solidification and cooling, as well as to interpret the subsequent microstructural changes occurring during HIP'ing.

4.1 RESULTS

4.1.1 Ti-54%Al Composition

The geometry of the as-received Ti-54%Al powder is illustrated in Figure 64. The powder is predominantly spherical, with numerous satellites, elongated and fractured particles. The satellites may result from partially solidified particles colliding during atomization. A higher percentage of fractured particles may be present due to the extensive handling of this sample. The interior of a fractured particle is shown in Figure 65. The essentially featureless outer perimeter of the fracture surface is likely due to handling damage. Within the fracture the dendritic structure is visible, along with several larger spherical features, which may originally have been individually atomized particles which were surrounded by larger molten droplets during atomization and solidification.

The solidification microstructure of the as-received Ti-54%Al powder is illustrated in Figure 66. Each particle consists of many grains of $\approx 45 \mu\text{m}$. The dendrite regions are clearly distinguished and range from 4 to $30 \mu\text{m}$ section thickness. A higher magnification

of these dendrite regions is shown in Figure 67, in which many dendrites can be seen to have a lamellar structure.

Many powder particles contained voids, such as shown in Figure 68. These voids could be due to either entrapped gas or solidification shrinkage. Although TGA powder particle pores have been attributed to entrapped atomizing gas ²⁴, judging by the morphology of the voids (i.e. not spherical) it is probable that most are due to solidification shrinkage.

4.1.2 Ti-48%Al-2%W Composition

Figure 69 illustrates the geometry of the Ti-48%Al-2%W powder in the as-received state. In comparison to the Ti-54%Al powder of Figure 64, it is evident that a higher proportion of small particles of Ti-48%Al-2%W exist, in agreement with the size distribution of Figure 52. The powder sample contains numerous satellites, irregularly shaped and broken particles. Although overall, fewer broken particles were present compared to the Ti-54%Al sample. This is consistent with the lower degree of handling experienced by the TiAl+W material.

The solidification microstructure of as-received particles is seen in the backscattered electron image of Figure 70. It is evident that the solidified microstructure consists of single phase primary dendritic regions surrounded by a two phase structure. The dendrite area fraction was found to vary between particles, but no consistent trend relating dendrite area fraction and particle size was apparent. The two phase structure exhibits a secondary dendritic structure with a secondary dendrite arm spacing of $\approx 6 \mu\text{m}$. The microhardness of the primary dendritic and secondary dendritic/interdendritic regions were measured as 47 and 33 Rockwell 'C' respectively.

The dendritic morphology is not well defined, but dendrites with orthogonal arms were observed, Figure 71. A feature of most dendritic areas was an etch surface relief

pattern of laths. Many dendrites are circumscribed by a thin phase, indicated in Figure 71, containing a lower concentration of tungsten as evidenced by the backscattered electron image of Figure 72.

The distributions of Ti, Al and W within the dark and light phases shown in Figure 72 are illustrated in Figure 73, in which the energy dispersive X-ray spectrum (EDS) for each phase is illustrated. It is apparent that for the backscattered image of Figure 72 most of the contrast is due to variations of the aluminum and tungsten concentrations. No evidence of a distinct tungsten enriched third phase was found.

Numerous Ti-48%Al-2%W particles contained voids such as shown in Figure 74. In contrast to those observed in the Ti-54%Al powder, most voids were located near the surface of the particles. Therefore, it is likely that these voids are due to entrapment of the atomizing gas during powder manufacture in agreement with the findings of reference ²⁴, with the remainder due to interdendritic shrinkage.

4.2 DISCUSSION OF POWDER MICROSTRUCTURE

As reviewed in section 2.1.1., considerable work has been directed at understanding the microstructural evolution of near γ -TiAl alloys during solidification and cooling at various rates. The aim of this work was to clarify the Ti-Al phase diagram and determine the kinetics for the various transformations involved. As such, it is of interest to rationalize the as-received microstructure of the present powder with the phase diagram of Figure 12, and the previously observed transformations occurring during rapid solidification.

4.2.1 Ti-54% Al Composition

The Ti-54% Al powder microstructure of Figure 67 corresponds to the microstructural evolution proposed in Reference 32, which is:

$$L \rightarrow [\alpha] + L \rightarrow [\alpha] + \gamma \rightarrow [\alpha + \gamma_c] + \gamma \rightarrow [\alpha_2 + \gamma_c] + \gamma \rightarrow [\alpha_2 + (\alpha_2 + \gamma) + \gamma_c] + \gamma \dots (18) .$$

The γ_c refers to a cellular morphology of γ which has been observed at dendritic boundaries, but was not seen in the Ti-54%Al powder. Accordingly, solidification proceeds by the formation of primary α dendrites, which with further cooling are surrounded by γ segregate. The α dendrites order to α_2 -Ti₃Al at about 1125°C, which contains multiple stacking faults¹⁴. The α_2 stacking faults act as nuclei for the growth of γ plates as described in¹¹⁷, and this leads to the lamellar structure seen in Figure 67. At sufficiently high cooling rates the decomposition of α_2 into $\alpha_2 + \gamma$ laths can be arrested, resulting in retained α_2 . According to the results of¹¹, for rapidly solidified powder the amount of retained α_2 is only significant (>1%) in particles <100 μm size. Nevertheless, in this TGA Ti-54%Al (for which all particles were >105 μm , see Figure 52) dendritic regions that do not exhibit a fully developed lamellar structure are evident, Figure 67. This would seem to indicate a faster solidification and cooling rate for the TGA powder compared to the powder of reference 31.

4.2.2 Ti-48%Al-2%W Composition

The presence of well defined primary dendrites in this powder (Figure 70) is a significant result, since in gas atomized powder of Ti-48%Al, Ti-48%Al-3%Cr and Ti-48%Al-2%Cr-2%Nb no dendritic areas were observed^{200,201}. It is possible that segregation of tungsten is responsible for the dendritic structure. Alternately, the thermal history during powder solidification of the various compositions may be different. This latter explanation seems less likely, since all these powder compositions were produced by the same manufacturer on the same equipment.

The presence of dendrites with orthogonal arms (Figure 71) indicates that these dendrites solidified with a cubic structure, consistent with the β -Ti region of the phase diagram. This agrees with the findings of¹⁴⁶, in which it was concluded that for binary TiAl powders, α dendrites clearly exhibited a hexagonal symmetry, but the symmetry of

β dendrites was "indistinct". As the powder particle cools the peritectic reaction $\beta + L \rightarrow \alpha$ can occur, thus forming a 'peritectic wall' of α (α_p) around some of the β dendrites, such as observed in Figure 71 and 72. It might be expected that the peritectic reaction could only occur in the larger particles corresponding to slower cooling rates, but this did not appear to be the case. However, it has been suggested that at moderate supercoolings recalescence occurs, thus providing a greater opportunity for β dendrites to be surrounded by peritectic α_p ⁶¹.

During equilibrium cooling the β dendrites transform to α , order to α_2 and decompose to $\alpha_2 + \gamma$ lamellae ³⁴. The micrograph of Figure 71 clearly indicates that these reactions do not occur during cooling of gas atomized particles, since the dendrite regions do not exhibit a lamellar structure. The β surface relief pattern exhibited by most dendrites may be indicative of a martensitic structure. The martensitic transformation $\beta \rightarrow \alpha'$ (disordered martensitic α) has been observed in Ti₃Al based compositions and found to be very dependent on cooling rate ¹⁸. Particles with a martensitic structure resulting from a $L + \beta \rightarrow \alpha'$ transformation have also been observed in Ti-47%Al produced by the plasma rotating electrode process ²⁰². It may be that martensitic α' is favoured in tungsten ternary alloys, since tungsten may suppress the diffusion required for other forms of transformation. This is consistent with the segregation of tungsten to β dendrites (Figure 73) during powder atomization, which occurs since the dendrites are the first phase to solidify from the melt. The increased tungsten concentration and the martensitic structure results in the higher microhardness (47 versus 33 Rockwell 'C') of the β dendrites. Thus, in Ti-48% Al-2% W powder it appears that a $\beta \rightarrow \alpha' + \alpha$ transformation can occur. The higher hardness for the martensitic areas evolved from β has been observed in other near γ -TiAl powder metallurgically produced powders also ²⁰².

Continued cooling of dendritic areas causes the ordering reaction $\alpha' + \alpha \rightarrow \alpha' + \alpha_2$. It appears that the cooling rate was sufficient to suppress the decomposition of $\alpha_2 \rightarrow \alpha_2 + \gamma$ lamellae, which was observed in many dendrites of the Ti-54% Al powder. This is a noteworthy result since both powder compositions were produced in the same manner - with

presumably similar cooling rates - it is indicative that the $\alpha_2 \rightarrow \alpha_2 + \gamma$ decomposition is affected by either the aluminum content, the tungsten ternary addition or martensitic structure of the prior β dendrites. The presence of ternary tungsten is probably the most important since the low diffusivity of tungsten solute retards diffusion assisted mechanisms¹¹⁷, thus promoting martensitic transformations.

It is interesting that in Figure 71 the peritectic wall is still discernable at room temperature, as it might be expected that the α' martensitic plates would extend through the peritectic layer. A compositional difference between the α' and peritectic α_p exists, Figure 72, with the α' containing a higher proportion of the heavier elements (i.e. tungsten). This compositional change could affect the $\alpha \rightarrow \alpha'$ martensitic reaction. Additionally, since the α_p peritectic wall forms at a lower temperature than the primary dendritic β , a lower internal strain energy should exist within the peritectic α_p , thus reducing the driving force for the martensitic reaction resulting in the retention of the peritectic wall to room temperature.

The interdendritic structure contains two phases (Figure 71 and 72). This is in contrast to that expected during equilibrium cooling, which would result in α interdendritic segregate, ordering to α_2 and then decomposing to $\alpha_2 + \gamma$ lamellae. The two phase structure of Figure 71 could form during the recalescence referred to previously in the discussion regarding the formation of the peritectic wall. During this recalescence a second nucleation event could occur, corresponding to formation of α secondary dendrites. Such a second nucleation event has been inferred for binary Ti-45%Al and observed thermally during controlled cooling of near γ binary TiAl¹¹⁸. Alternately, the depletion of titanium from the liquid, due to the formation of β dendrites, allows α to nucleate directly from the aluminum enriched liquid in accordance with the phase diagram. Thus, in the regions between the primary β dendrites, the solidification first forms secondary α dendrites. Subsequently, with further aluminum enrichment of the remaining liquid, γ segregate solidifies between the secondary α dendrites. The peritectic reaction $L + \alpha \rightarrow \gamma$ is not observed due to the very slow kinetics of the $\alpha \rightarrow \gamma$ reaction¹¹⁹.

The microstructural evolution during solidification and cooling of Ti-48%Al-2%W is thus summarized as:

where; α_p is the α phase of the peritectic wall,

α' is disordered martensitic α phase,

α_s is secondary dendritic α ,

γ_{seg} is the γ phase segregate, and

the subscript 2 indicates the ordered version of the various α phases.

Note that in equation 2, the $\beta \rightarrow \alpha'$ transformation is shown to occur before the solidification of α_s . In fact, it is not clear from the current results if this martensitic reaction occurs before or after the formation of α_s or α_p .

5. HOT ISOSTATIC PRESSING

5.1 MICROSTRUCTURAL RESULTS

5.1.1 Ti-54%Al Composition

The limited amount of Ti-54%Al powder available allowed evaluation of only two HIP conditions, 1025°C and 1100°C at 200 MPa for 2 hours. The microstructures resulting from HIP'ing at 1025°C and 1100°C are shown in Figures 75 and 76 respectively. The grain structure resulting from both HIP conditions is similar and consists of regions of very fine grains ($\approx 6 \mu\text{m}$ size) and coarse grains (50-70 μm size), with a few recrystallization twins present.

In both HIP'ed conditions the prior particle boundaries are clearly discernable. As alluded to previously, this is probably due to the extensive handling, and consequent contamination, of the Ti-54%Al sample. Porosity is present in the material HIP'ed at 1025°C (Figure 75). Porosity completely contained within the prior particles (Figure 75a) probably corresponds to solidification shrinkage of the type illustrated in Figure 68. As well, pores exist at prior particle boundaries (PPB's), Figure 75b. Both these features are indicative of insufficient metal flow for complete densification at the 1025°C HIP temperature. In the sample HIP'ed at 1100°C (Figure 76) no porosity could be found.

After HIP'ing at 1025°C some coring is evident, Figure 75a. This is more prevalent in the larger prior particles, in which solidification was slow enough for local concentration gradients to develop during powder manufacture. Coring is not observed in

the material HIP'ed at 1100°C (Figure 76) since the higher temperature has allowed sufficient diffusion to occur to eliminate local concentration gradients.

5.1.2 Ti-48%Al-2%W Composition

This composition was subjected to four HIP cycles. For each cycle the maximum pressure available (200 MPa) was always applied and the time at maximum pressure and temperature was always two hours. Due to the observation of porosity in the Ti-54%Al sample HIP'ed at 1025°C, the minimum HIP temperature applied to the ternary composition was 1050°C. The other HIP temperatures were: 1100°C, 1150°C and 1250°C. This temperature range spans the α/α_2 ordering temperature of 1125°C.

In the microstructures of all the HIP'ed conditions, the primary dendrite regions observed in the solidified powder (see Figure 70) are still clearly discernable, as seen in Figure 77. Also evident, is non-uniformity of the spatial distribution of these dendrite regions and a marked change in the local microstructure between prior particles. However, the PPB's are not decorated by any oxide or second phase particles, as was the case for HIP'ed Ti-54%Al powder. Furthermore, compositional analysis by glow discharge mass spectroscopy of the HIP'ed compacts, see Table XIII, indicated carbon and oxygen contents within 10% of the respective content in the starting powder (see Table XI). Thus, the reduced handling and protective argon environment of the Ti-48%Al-2%W powder has resulted in a 'cleaner' product. The variation in the nitrogen and iron contents between the starting powder (Table XI) and HIP'ed samples (Table XIII) may be attributable to differences in the calibration of the spectrometers used for the two analyses. (The glow discharge mass spectrometer used for analysis of HIP'ed samples cannot analyse powder. Thus, direct comparison of analysis from both spectrometers on the same material was not possible.)

A small amount of porosity could be found in the powder HIP'ed at 1050°C. As seen in Figure 77, this porosity appeared to be located at PPB's. Porosity was not observed in the metallographic sections of material HIP'ed at greater than 1100°C.

The overall microstructure of material HIP consolidated at each of the four temperatures is illustrated in Figure 78. Common to each of the HIP temperatures, but especially evident at the lower HIP temperatures, is microstructural inhomogeneity between prior primary dendrites and interdendritic regions. The observations resulting from detailed examination of these two regions is summarized in the next two sections. For simplicity, the primary dendritic areas of the powder microstructure will be referred to as prior dendrites and the area between prior dendrites (including the secondary dendrites) will be referred to as interdendritic regions. This examination concentrates on the highest (1250°C) and lowest (1050°C) HIP'ed conditions.

5.1.2.1 Interdendritic Regions

Figure 79 shows higher magnification optical and SEM micrographs of the 1250°C HIP'ed material. One of the most notable features of Figure 79 is the lamellar appearance of some grains. However, in the backscattered electron image (BEI) of an unetched region (Figure 79d) this lamellar feature is not observed. Furthermore, the energy dispersive X-ray spectrum (EDS) of the the light and dark lamellae of Figure 79b are identical, see Figure 80, and are nearly identical to the γ spectrum of Figure 73. Thus it is concluded that the lamellar feature results from localized chemical attack during etching. This conclusion is supported by references **203,204**, where similar effects were observed.

Aside from the lamellar feature, three distinct phases are present in the micrographs of Figure 79c. The major phase exists as equiaxed grains with a mean size of $\approx 13 \mu\text{m}$. From the EDS analysis of the equiaxed grains, shown in Figure 81a, they are concluded to be γ phase. The second phase of Figure 79c exhibits an equiaxed morphology also, but is generally smaller than the γ grains. This phase images brighter in the

backscattered electron images, indicating higher levels of tungsten and titanium than the γ phase. Comparing the EDS analysis of this phase (Figure 81b) with the γ phase (Figure 81a), confirms the higher tungsten and titanium levels, as well as indicating a lower aluminum content. Thus, this second phase corresponds to α_2 . The third phase of Figure 79c consists of particles that are either spherical ($\approx 3 \mu\text{m}$ size), or elongated with dimensions of $\approx 2 \mu\text{m}$ by $10 \mu\text{m}$. The spherical particles are typically found within γ grains, while the latter morphology is usually located at triple points and grain boundaries. EDS analysis of this third phase, Figure 81c, indicates significant enrichment with tungsten. Unfortunately, since this phase resisted thinning during jet-polishing, even at the maximum TEM accelerating voltage, a diffraction pattern to determine the crystallographic structure could not be obtained.

The interdendritic microstructural details of 1250°C HIP'ed material are shown in the TEM micrograph of Figure 82. Predominantly equiaxed recrystallized grains with numerous recrystallization twins exist. A few grains contain a dislocation substructure. Overall, the dislocation density is low. The presence of the dislocation substructure and recrystallized twins gives a dislocation free path or 'effective grain size' of $\approx 4.9 \mu\text{m}$. Using the diffraction contrast imaging procedure outlined in the previous chapter, the dislocations present were identified as predominantly $\frac{1}{2}\langle 110 \rangle$ ordinary dislocations, with a minor quantity of $\frac{1}{2}\langle 112 \rangle$ and $\langle 101 \rangle$ superdislocations present.

The interdendritic microstructure resulting from HIP'ing at 1050°C is illustrated in Figure 83. The notable differences between this structure and the 1250°C HIP'ed condition are the presence of deformation twins (which are typically very narrow), absence of recrystallization twins (which are usually much wider than deformation twins), well developed dislocation substructure and a grain size of $\approx 5 \mu\text{m}$. The well developed substructure of mechanical twins and subgrain boundaries has an 'effective grain size' of $\approx 2.4 \mu\text{m}$. Diffraction contrast imaging experiments indicated that the predominant dislocations in the interdendritic region of 1050°C HIP'ed material are $\frac{1}{2}\langle 110 \rangle$. Some super-

dislocations are present, but due to sample tilting limitations in the TEM they could not be identified.

The etch contrast of the optical micrograph of the 1050°C HIP'ed material, Figure 78 is due to local composition gradients retained from the solidified condition. This can be seen by the SEM backscattered electron image of Figure 84, in which a secondary dendritic structure, delineated by contrast differences, similar to that of Figure 70 is still apparent. Thus, the secondary dendritic structure of the powder microstructure is retained during HIP'ing at 1050°C.

Unlike the 1250°C HIP'ed material, distinct single phase grains of α_1 do not exist after HIP'ing at 1050°C. The structure predominantly consists of γ grains with dislocations and deformation twins, Figure 85. The selected area diffraction pattern of Figure 85b indicates a minor presence of α_2 , which in agreement with Reference 22 forms preferentially along the γ twin boundaries. Many γ/α_2 interfaces and deformation twin boundaries are decorated with precipitates or larger third phase particles, Figure 86. SEM backscattered imaging revealed tungsten enriched bands, Figure 87, corresponding to the interfaces of Figure 85a, and larger (up to $\approx 1.5\mu\text{m}$) tungsten rich particles. The small size of the tungsten rich phase in this HIP'ed condition precluded EDS analysis.

After HIP'ing at 1100°C, a recrystallized structure is observed in some areas, Figure 88. It is apparent that grain boundary migration from a dislocation free recrystallized region to an unrecrystallized region with a high dislocation density occurs. The migrating grain boundary can be pinned by the third phase. At this HIP temperature, the formation of recrystallization twins occurs, often developing boundaries with a ledge structure, Figure 89, in agreement with previous observations⁴¹. In this HIP'ed condition the mean size of the dislocation free recrystallized grains is $\sim 6.4\mu\text{m}$, while unrecrystallized areas have an effective grain size of $\sim 2.6\mu\text{m}$. The dislocations in this condition, Figure 90, were found to be predominantly $\frac{1}{2}\langle 110 \rangle$ ordinary dislocations, with limited

$\frac{1}{2}\langle 112 \rangle$ and $\langle 101 \rangle$ superdislocation activity. This dislocation activity is consistent with that discussed in section 2.2.

The grain size and effective grain size measurements, mentioned in the preceding paragraphs, for each of the HIP'ed conditions are summarized in Figure 91.

5.1.2.2 Prior Dendritic Regions

Figure 92 shows that the prior dendrite regions contain three well defined phases, together forming a fine grained equiaxed dislocation free structure. The grain size within these prior dendritic areas increases with HIP temperature as illustrated in Figure 93. As well, image analysis of optical micrographs indicated that the prior dendritic area fraction decreases with increasing HIP temperature.

The nature of the three phases within the prior dendrites were determined by EDS analysis. The EDS analyses corresponding to each phase identified in Figure 92a are illustrated in Figure 94. The spectrum for the phase labelled A1 in Figure 92 contains high levels of tungsten. The regions A2 and A3 correspond respectively to the spectrums for the α_2 and γ phases shown in Figures 81a and 81b, except for a slightly higher level of tungsten.

Optical micrographs of the prior dendrite regions are shown in Figure 95. Some of the prior dendrite regions of the material HIP'ed at 1050°C exhibit three distinct regions, consisting of an inner core surrounded by a lamellar structure and an outer perimeter of fine second phase particles. This is in contrast to the optical micrograph of the 1250°C HIP'ed material, which shows a three phase structure similar to the backscatter and TEM micrographs of Figure 92. Many dendrites in the 1050°C HIP'ed condition are surrounded by coarse grains (up to 40 μm). Figure 78.

5.2 HIP'ED MECHANICAL PROPERTIES

5.2.1 Hardness Results

The hardness of samples of both compositions HIP'ed at each temperature are plotted in Figure 96. For both compositions the hardness decreases with increasing HIP temperature. This can be attributed to the larger grain size obtained at higher HIP temperatures (see Figure 91). It is also clear that the single phase γ -Ti-54% Al composition has a lower hardness than the TiAl+W which may be due to differences in aluminum or tungsten content.

The microhardness of the prior dendritic and interdendritic regions is compared in Figure 97 for the TiAl+W composition. The most notable result is that in all conditions the dendritic or prior dendritic regions have a higher hardness. Also, there is a significant decrease in microhardness resulting from HIP'ing at 1250°C. The difference between the microhardness of interdendritic and prior dendritic regions remains virtually constant for all HIP temperatures, suggesting that minimal long range homogenization occurs during HIP'ing.

5.2.2 Tensile Results

The room temperature tensile results are listed in Table XIV, with examples of the stress-strain curves illustrated in Figure 98. The best room temperature ductility attained is 1.5% for the sample HIP'ed at 1250°C, which is better than the room temperature ductility reported in Table VIII for a similar alloy also prepared from powder. As such it is felt that the attention to cleanliness during the encapsulation of powders was successful in preventing contamination of the metal.

Several of the room temperature tensile samples failed to reach a 0.2% yield point due to lack of ductility and no sample showed any signs of necking. Even the fracture surface of the most ductile sample (1.5% elongation) shows features characteristic of

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PM-1 3½"x4" PHOTOGRAPHIC MICROCOPY TARGET
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brittle failure, see Figure 99. The region of fibrous failure is small and no circumferential 'shear lip' is present. A radial cracking zone (not originating from the specimen centre) exists for most of the failure surface and is indicative of rapid unstable crack growth^{26,205}. This result exemplifies the brittleness of TiAl intermetallics at room temperature.

Voids remaining after HIP'ing lead to premature failure as evidenced by Figure 100, in which failure occurred in the sample shoulder radius due to the presence of a large void, creating a local stress concentration. This observation illustrates the importance, but difficulty, of attaining complete densification.

The results of elevated temperature tensile tests are given in Table XV. As seen in Figure 101, duplicate test coupons gave excellent repeatability. This was typical of tests at all temperatures. The tensile failure surface at ambient temperature and 550°C is predominantly transgranular and very flat, Figure 102. In contrast, at the 850°C test temperature, tensile failure occurs along primarily along PPB's, as well as grain boundaries, Figure 103.

5.3 TENSILE DEFORMED MICROSTRUCTURES

The tensile deformed microstructures of the 1050°C and 1100°C HIP'ed conditions, tested at room temperature and 550°C, are characterized by a very non-uniform density of dislocations and deformation twins, Figure 104. Ostensibly deformation free grains exist adjacent to heavily deformed grains, with the development of a dislocation sub-grain structure evident. Although still localized, deformation was more uniform at the 850°C test temperature. The dislocations present after tensile testing of 1050°C and 1100°C HIP'ed material at all temperatures are predominantly $\frac{1}{2}\langle 110 \rangle$ ordinary dislocations with very few superdislocations visible.

Material HIP'ed at 1250°C exhibits a considerably more uniform structure of deformation twins and dislocations after tensile testing, Figure 105. Deformation twins

containing arrays of the $1/6\langle 112 \rangle$ twinning partial dislocation crossing recrystallization twins exist, as seen in Figure 106. The development of a deformation twin from $1/6\langle 112 \rangle$ partial dislocation array is illustrated in Figure 107.

After tensile deformation of 1250°C HIP'ed material, isolated dislocation free grains exist, Figure 108. SAD indicated that some dislocation free grains are α_2 , consistent with the backscattered image of Figure 79c. The α_2 and adjacent γ grains have the $(0001)_{\alpha_2} \parallel \{111\}_{\gamma}$ and $\langle \bar{1}\bar{1}20 \rangle_{\alpha_2} \parallel \langle \bar{1}10 \rangle_{\gamma}$ orientation relationship. In the 1250°C HIP'ed material tensile tested at 850°C, small dislocation free convex shaped grains exist at triple points and grain boundaries, Figure 109. Unfortunately, their small size makes structure determination by SAD uncertain.

The dislocations present in 1250°C HIP'ed material after tensile testing at ambient temperature and 550°C are predominantly $1/2\langle 110 \rangle$ ordinary dislocations, with very few superdislocations present. This is similar to material HIP'ed at the lower temperatures. After tensile testing at 850°C the density of dislocations and deformation twins is increased. Additionally, arrays of $1/2\langle 112 \rangle$ superdislocations were identified, Figure 110, however the $1/2\langle 110 \rangle$ ordinary dislocations are still predominant.

5.4 DISCUSSION OF HOT ISOSTATIC PRESSING

5.4.1 Ti-54%Al Composition

The coarse grained (50-70 μm) regions of Figures 75 and 76 probably result from grain growth during HIP'ing, since the grain size in the powder is $\approx 45\mu\text{m}$. However, it appears that the fine grained regions result from local dynamic recrystallization during HIP'ing. The driving force for localized recrystallization could be either the higher internal strain energy of some powder particles resulting from solidification, or variations in the local deformation during HIP'ing. It would be expected that smaller particles would contain a higher level of internal strain (due to faster solidification) and therefore recrystallization would be more evident in these particles. However, in the micrograph of

Figure 75 the PPB's are clearly discernable and it can be seen that recrystallization is not limited to the finer particles. As such, it is probable that the recrystallization results from local deformation during HIP'ing.

5.4.2 Ti-48Al%-2%W Composition

The observation of porosity in 1050°C HIP'ed material (Figure 77) indicates that the HIP temperature required for consolidation is at least 1100°C, although as evidenced in Figure 100 isolated porosity may still exist. This result is in agreement with the density measurements of Figure 20 and 21.

The non-uniform spatial distribution of dendrite regions in the HIP'ed condition (Figure 77) corresponds to the varying area fraction of dendrites in individual powder particles (Figure 70). This variation in turn causes differing amounts of α (secondary α) and γ_{seg} (γ phase segregate) between particles due to the partitioning of tungsten to dendrites during powder atomization (Figure 73). As such, some PPB's can be located by the marked change in microstructure.

The irregular distribution of dendrites, and the ensuing tungsten partitioning, among powder particles (Figure 70) is also responsible for the microstructural inhomogeneity in HIP'ed material evident in Figure 78. Thus, for all HIP'ed conditions the microstructure can be divided into well defined prior dendritic and interdendritic regions. In the latter regions, recrystallization proceeds to a greater extent with increasing HIP temperature, resulting in a coarser grain structure, Figure 91. Moreover, at the higher HIP temperatures, recrystallization of the interdendritic regions consumes the outer periphery of the prior dendrites, thus lowering the area fraction of prior dendrites, Figure 96. This contributes to improved microstructural homogeneity resulting from the higher HIP temperatures.

Prior dendritic regions contain fine equiaxed dislocation free grains, which increase in size with the HIP temperature, Figure 96. Due to the segregation of tungsten to dendrites during solidification, the areas surrounding the prior dendrites have a lowered tungsten concentration, allowing faster grain growth during exposure to the HIP temperatures, thus creating the coarse grained regions observed around many prior dendrites, Figure 77. Thus, the coarse grained regions coincide with prior particles that contained large dendrite area fractions. A structure of fine and coarse grained regions has been reported previously, but the nature of its origin was not determined ⁶⁸, and exemplifies the importance of a uniform microstructure in all the powder particles. Important differences in the deformation behaviour of the prior and interdendritic regions are discussed in a later section.

5.4.2.1 Interdendritic Microstructural Evolution During HIP'ing

It is postulated that the α_2 in the as-received powder transforms to γ during the HIP cycle heat up, as discussed in section 2.3.2.3.1b ⁶⁵. The increase in HIP temperature and pressure shown in Figure 53 is consistent with the occurrence of such a transformation. However, since at the peak HIP temperature or during cooling from the maximum HIP temperature the α/α_2 reforms (as discussed in the next paragraph), the occurrence of the $\alpha_2 \rightarrow \gamma$ reaction could not be confirmed.

The morphology of the α_2 that reforms during HIP'ing is dependent on the HIP temperature. After HIP'ing at 1050°C, α_2/γ lamellae exist along deformation twins (Figure 85). The formation of α_2 lamellae along twin boundaries has been previously observed (see section 2.1 and Figure 7). The recrystallization that occurs during 1250°C HIP'ing results in single phase grains of α_2 (Figure 79c and 109). Interestingly, these single phase grains of α_2 maintain the $(0001)_{\alpha_2} \parallel \{111\}_{\gamma}$ or $\langle \bar{1}\bar{1}20 \rangle_{\alpha_2} \parallel \langle \bar{1}10 \rangle_{\gamma}$ crystallographic relationship with adjacent γ grains common to the α_2/γ lamellae. Thus, it seems likely that these recrystallized α_2 grains form from α_2 lamellae that may develop along

recrystallization twins at the 1250°C HIP temperature. The higher area fraction of α_2 after HIP'ing at 1250°C is consistent with the phase diagram (Figure 12).

An important reaction occurring during HIP'ing is the formation of a tungsten rich third phase. A tungsten enriched third phase has been observed previously for ternary TiAl+W^{117,145} and concluded to be body centred cubic β phase. Although for the current material the crystallographic structure of this phase could not be determined, the EDX spectrum of Figure 81c is consistent with the probable Ti_2AlW composition of β . Thus, the third phase in this alloy is concluded to be β .

During HIP'ing at 1050°C, tungsten diffuses to α_2/γ lamellae interfaces and deformation twins, causing the precipitation of the β phase. Hence, very fine β precipitates along these interfaces exist (Figure 86b). The α_2 interfaces may be a more favourable location for β phase nucleation due to the higher tungsten content of α compared to the γ phase. As such, larger β particles are sometimes observed associated with α_2 lamellae (Figure 86a). This β/α_2 interaction has been observed previously in a TiAl+Cr ternary alloy²¹⁶.

At higher HIP temperatures the kinetics of β formation are increased and so larger β particles are observed. In the 1250°C HIP'ed material β is predominantly observed along recrystallized grain boundaries and triple points (Figure 79c). Thus, it would appear that recrystallization occurred prior to β formation. This is in contrast to the process occurring during HIP'ing at 1100°C, in which β particles are observed to pin migrating grain boundaries during recrystallization (Figure 88). These results imply that the kinetics of β formation and recrystallization are very dependent on the HIP temperature. As such, in the 1250°C HIP'ed material β formation is initiated by tungsten diffusion to grain boundaries, rather than lamellae interfaces or deformation twins, since both these features are annihilated during recrystallization.

From the preceeding results, it is concluded that during HIP'ing at 1250°C, the interdendritic regions of the powder recrystallize and third phase β develops. Subsequent cooling from the HIP temperature causes ordering of the α into α_2 . Thus, the microstructural reactions occurring in the interdendritic regions during HIP'ing can be summarized as;

5.4.2.2 Prior Dendrite Microstructural Evolution During HIP'ing

HIP'ing at 1250°C transforms the primary dendritic α_2' into the equiaxed structure of γ , α_2 and β , seen in Figure 92. However, it is not known if the martensitic α_2' first transforms to γ , as outlined in section 2.3.2.3.1b and in accordance with reference 85, but, as before, the increase in HIP temperature and pressure is consistent with such a transformation. This structure is similar to that existing around the perimeter of some dendrites in the 1050°C condition, Figure 98. Thus, it is postulated that at a 1050°C HIP temperature, insufficient time and temperature are available to complete the transformation to an equiaxed structure. Also, it would appear that the transformation begins at the dendrite perimeter and proceeds toward the centre of the dendrite. These transformations may occur either because coring that exists in the solidified dendrites is homogenized or the α_2' martensitic structure that exists within the solidified dendrites is tempered (see Figure 71). In neither of the HIP'ed conditions is there any evidence of the α_2 peritectic wall which was present around some of the dendrites in the as-received powder.

Consistent with the partitioning of tungsten to dendrites during powder solidification, the EDS results for all three phases in prior dendritic regions (Figure 97) indicates a slightly higher tungsten concentration compared to the interdendritic regions (Figure 81).

From the preceeding discussion, the microstructural transformation occurring in the prior dendritic regions during HIP'ing can be summarized as:

This reaction proceeds to a greater degree at higher HIP temperatures.

The lower dislocation density in the prior dendritic regions (Figure 92) implies that these areas deform minimally during HIP'ing. This is consistent with the higher microhardness and in turn, higher flow stress of prior dendritic areas (Figure 97), which may be due to two factors. First, the very fine microstructure and the increased concentration of tungsten may cause solid solution hardening. Second, the higher volume fraction of β phase in the prior dendritic regions may contribute to increased hardness. In cast and stabilized TiAl+3%Cr, β phase was found to have a higher hardness than either the α_2 or γ phases²⁰⁶. (Unfortunately, in the current work, even using the lightest microhardness indenter load, the hardness of the individual β particles could not be determined.) It is concluded, then, that the flow stress of the dendritic regions is higher than the surrounding metal. As such, it would be expected that the deformation required for consolidation during HIP'ing will occur predominantly in the interdendritic regions.

In the interdendritic regions the recrystallized structure (see Figure 82) existing after HIP'ing at 1250°C will lower the internal strain level, thus lowering the microhardness in this condition. In dendritic areas, significant coarsening of the three phase equiaxed structure occurs during HIP'ing at 1250°C, Figure 96, decreases the local microhardness.

5.4.3 TENSILE PROPERTIES

The low strength and ductility of the Ti-54%Al sample could be due to several factors. Firstly, after HIP'ing, this composition consists of single phase γ which, consistent with the hardness results, exhibits low strength and ductility compared to the duplex

structure. Secondly, as seen in Figure 75, the Ti-54%Al tensile sample HIP'ed at 1025°C was not fully dense. It is possible that premature tensile failure was initiated by internal porosity. Lastly, due to the considerable 'handling contamination' of the Ti-54%Al powder sample (see section 3.2) oxides lined the PPB's, see Figure 75. This contamination could account for premature failure and such a situation lends credence to the conclusion of the next paragraph regarding powder cleanliness during encapsulation.

For the ternary alloy, the attainment of a room temperature ductility of 1.5% is a significant result for this composition. As outlined in section 2.4.1, the highest tensile ductilities are usually obtained for duplex microstructures consisting of $\approx 30\%$ $\alpha_2 + \gamma$ lamellae ²¹ (see Figure 8). Such duplex structures exhibit room temperature tensile ductility of $\approx 1.8\%$ (see Table IV). In the current TiAl+W composition, $\alpha_2 + \gamma$ lamellae were not observed and a microstructure of single phase α_2 and γ grains with third phase β exists (see Figure 79c). Nevertheless, the room temperature ductility of 1.5% is nearly equivalent to the 30% lamellar structure. Furthermore, the alloy of Table IV contains Cr and Mn, both of which have a ductilizing effect (see section 2.5), while W apparently reduces the room temperature ductility ². Thus it might be expected that the ductility of the TiAl+W composition investigated would be significantly lower than 1.5%. This improved ductility may be due to the presence of the blocky β phase predominantly located at triple points and along grain boundaries. For Cr ternary alloys, it was concluded that β phase formation along grain boundaries was necessary for improving hot workability, (see section 2.5.1). The ductility values attained also indicate that powder cleanliness was maintained during encapsulation and HIP'ing.

The most prominent result of the elevated temperature tensile tests is the lack of a pronounced brittle-ductile transition at $\approx 700^\circ\text{C}$ as observed for many other near γ -TiAl alloys. However, this result is in agreement with the results of Table IV. The highest ductility obtained was 4.7% at 850°C. In contrast, many binary and ternary near γ -TiAl compositions exhibit tensile strains of up to 40% when tested above 700°C.

The effect on the fracture strength, as a function of HIP temperature, of increasing the test temperature is illustrated in Figure 111. It is evident that at the two lower test temperatures the effect of HIP temperature on fracture strength is similar to that of Figure 96 for the hardness. Hence, increases in the volume fraction of recrystallized grains, and the corresponding decrease in the dislocation substructure, as a result of HIP'ing at higher temperatures reduces the strength. These two trends produce an increasing effective grain size with HIP temperature. Thus, the strength decrease with increased HIP temperature could be due to a Hall-Petch effect. The fine β phase (particles of $<1.5 \mu\text{m}$ in Figure 86) of the 1050°C HIP'ed condition may also contribute to strengthening.

At 850°C the fracture strength was almost independent of the HIP temperature. This difference corresponds to the change in the fracture path from transgranular at the lower two temperatures, Figure 102, to PPB's and intergranular at 850°C , Figure 103. This result indicates that despite the cleanliness of the PPB's, they still represent a potential failure path.

In the 1050°C and 1100°C HIP'ed conditions, the non-uniform deformation structure (Figure 104) corresponds to the microstructure (Figure 78). The degree of deformation within each grain may be related to its crystallographic orientation with respect to the loading direction. Thus, in these HIP'ed conditions a small volume fraction of the material contributes to the accommodation of deformation, with potentially large strain discontinuities at grain boundaries. Consequently, modest tensile elongation will cause significant local internal strains, resulting in poor ductility. At the 850°C test temperature deformation is more uniform and thus higher ductilities are obtained.

In a similar manner to the previous paragraph, the more uniform deformation of 1250°C HIP'ed material contributes to the attainment of higher ductility. For this HIP'ed condition, the improved ductility at 850°C test temperature is due to the increased $\frac{1}{2}\langle 110 \rangle$ dislocation activity and deformation twinning, with a possible minor contribution due to

$\frac{1}{2}\langle 112 \rangle$ superdislocation motion. It is speculated that during tensile testing at 850°C incipient recrystallization may occur, as evidenced by small dislocation free grains (Figure 109). Deformation assisted recrystallization is known to occur during compressive straining of near γ -TiAl alloys^{86,87}. However, the phase existing within these grains could not be established. Therefore, it is also possible that they are β resulting from HIP'ing as outlined previously.

6. ISOTHERMAL COMPRESSION

6.1 RESULTS

6.1.1 Macroscopic Deformation Behaviour

The deformed shape of the cylindrical compression samples is illustrated in Figure 112. The grooves machined in the end faces of the sample (see Figure 58) are clearly visible and have radially expanded uniformly. The proximity of the largest groove to the diameter of the deformed sample indicates that lubrication of the sample-platen interface with the glass frit/boron nitride mixture was effective at reducing friction. Nevertheless, deformation is not homogeneous with 'dead metal' regions adjacent to each platen. The dead metal regions were most prominent in samples compressed at 900°C, Figure 113. These regions experience less deformation due to the additional constraint of sample-platen friction. The prior dendritic areas are easily visible in Figure 113 and remain almost undeformed at the sample-platen interface. One result of this constraint is the barreling of the sample sidewalls evident in Figure 113.

As a result of the inhomogeneous deformation and insufficient plastic capacity of the metal, cracking was observed in many samples. Three distinct types of cracks occurred. The most common were radial cracks emanating from the outer circumference of the sample, Figure 114. Note that in Figure 114 a total true strain of only 0.26 was applied. These cracks result from the inability of the metal to accommodate hoop stresses during deformation. A few incidents of axial centre line cracking was also observed. These cracks propagated from the top and bottom platen-sample interfaces through the complete thickness of the sample along its central axis, Figure 115. These cracks result

from radial tensile stresses associated with metal flow in the radial direction. Despite the presence of such a crack, residual hoop stresses retain the deformed sample shape, hence these cracks are only detected when the sample is cut in half, and the residual hoop stress eliminated. Cracking along PPB's was evident in one metallographic section, Figure 116. Note that the PPB crack of Figure 116 occurred after only 0.05 true strain. The incidence of the various types of cracks as a function of strain rate and temperature is summarized in Table XVI.

6.1.2 True Stress - True Strain Results

Isothermal compression tests were conducted at each of the temperature - strain rate combinations identified in Table XVI. A typical example of the load/specimen height data is illustrated in Figure 117. The maximum variability in the load data is ≈ 270 N or about 3.8%. The percentage load variability increased for samples tested at lower Zener-Hollomon parameters, due to the lower loads required. Almost no variability was observed in the specimen height measurements.

The load/specimen height data was transposed to true stress - true strain curves according to equations 16 and 17 of section 3.8. A complete listing of the resulting true stress - strain curves is given in Appendix C. From this data the four generic true stress - true strain curves of Figure 118 were identified. The unique attributes of each curve of Figure 118 are as follows:

Curve Type 1. Typical of tests at low temperature - high strain rate combinations exhibiting a high peak stress (>400 MPa) at large strains. With increasing strain, limited flow softening occurs.

Curve Type 2. As the temperature is increased the stress strain behaviour is characterized by a marked stress peak at low strain, followed by significant softening.

Curve Type 3. With further increased temperature or decreased strain rate curve 2 evolves to curve 3. A peak true stress at low strains still exists, but is followed by minimal flow softening. Consequently, a steady state flow stress is attained at low strain.

Curve Type 4. This stress-strain relationship results from compression at high temperatures and low strain rates. In this curve no peak true stress is observed. Rather, the flow stress slowly increases as more strain is applied.

Although the example curves of Figure 118 are all for 1050°C HIP'ed material, the true stress - strain results for 1250°C material can also be similarly described, except that the curve type 3 response was not observed. The temperature/strain rate combinations exhibiting each of the four curve types is summarized in Table XVI. The compression tests exhibited good repeatability as evidenced by Figure 119.

The effect of increased temperature or decreased strain rate on the compression behaviour is similar, see Figure 120. Both trends lower the peak true stress and decrease the amount of flow softening. Thus, at higher temperatures or slower strain rates, the strain required to attain a steady state flow stress is decreased.

Consistent differences in the true stress - strain curves of material HIP'ed at 1050°C and 1250°C are apparent, Figure 121. The 1250°C HIP'ed material always has a higher peak stress and exhibits less flow softening. At all strain rates, except 10^{-1} s^{-1} , at high strain levels ($\epsilon=0.65$), the flow stress of the two HIP'ed conditions is quite similar. For example, the flow stresses at $\epsilon=0.65$ for the 1050°C and 1250°C HIP'ed conditions after deformation at 1100°C and 10^{-3} s^{-1} are 79 MPa and 98 MPa respectively. At 10^{-1} s^{-1} the two HIP'ed conditions require much larger strains to converge to similar flow stresses, since the strain necessary to attain a steady state flow stress is increased.

6.1.3 Microstructural Changes During Compression

As shown in section 6.1, the deformation imposed in the compression samples is macroscopically inhomogeneous. Consequently, all microstructures illustrated are for areas located approximately along the sample central axis, at half the sample height. At this location sample-platen friction effects are minimized. All microstructures shown correspond to applied true strains of ≈ 0.8 , unless indicated otherwise.

An important change during isothermal compression is improved microstructural homogeneity. Even for the most inhomogeneous 1050°C HIP'ed condition (see Figure 78), compression to a true strain of 0.8 (~120% engineering strain) greatly improves microstructural homogeneity, Figure 122. Nevertheless, as seen in the micrographs of Figure 113 and 116, prior dendritic areas are still clearly discernable in all areas of the compressed samples. Furthermore, some PPB's could be delineated by a marked change in the density of β phase particles, Figure 123. The change in β phase density results from the segregation of tungsten to dendrites during powder solidification, as discussed in section 5.1.2.

Figure 123 also illustrates that for the 1050°C HIP'ed condition, the prior dendrites are seen to be made up of discrete β phase particles. Comparison to the as-HIP'ed prior dendritic region (Figure 92) indicates that the β particles coarsen considerably during isothermal compression. This occurs even for strain rates of 10^{-1} s^{-1} which involve short exposure to elevated temperature (see Figure 61). For 1250°C HIP'ed material, the β phase of the prior dendritic regions has also coarsened considerably during isothermal compression.

The deformed microstructures of each of the stress - strain curve types illustrated in Figure 118 exhibit unique characteristics which can be summarized as follows:

Curve Type 1. Deformation according to this stress - strain relationship results in a microstructure with a high dislocation density and many deformation twins, some of which are curved, Figure 124. Small local regions of recrystallized grains exist, as evidenced by the presence of small dislocation free grains. A significant amount of superdislocations activity occurs to accommodate the deformation as seen in Figure 125.

Curve Type 2. The curve 2 stress - strain relationship results in microstructures that exhibit a more homogeneous grain size, Figure 123a. In particular, the coarse grained regions surrounding the prior dendrites in the 1050°C HIP'ed condition (Figure 78) no longer exist. An almost completely recrystallized grain structure exists after 0.8 strain, with a low density of dislocations which are evenly distributed, Figure 126. As a result

of recrystallization, very few deformation twins exist, but recrystallization twins are present.

Curve Type 3. After compression corresponding to these stress - strain curves a coarser grain size exists, $>10\text{ }\mu\text{m}$ for the 1050°C HIP'ed material, Figure 127. This compares to an as-HIP'ed grain size of $\approx 5\text{ }\mu\text{m}$, see section 5.1.2.1. No 1250°C HIP'ed material displayed this type of stress strain relationship, see Table XVI.

Curve Type 4. This stress - strain behaviour results in the largest grains. In the 1050°C HIP'ed material, grains of up to $34\text{ }\mu\text{m}$ are present, with an average of $\approx 17.5\text{ }\mu\text{m}$, Figure 123b. In contrast to the microstructure resulting from curve type 2 compression, large grains surrounding prior dendritic regions are still present. Comparison of Figure 128 and 126 indicates that compression at the higher temperatures and slower strain rates results in significantly larger grains, that contain orderly dislocation arrays. Even after compression at 1200°C and 10^{-3} s^{-1} the prior dendritic regions are still discernable, Figure 129, because of the fine grained three phase dislocation free structure. The effect of applied strain on the grain size is shown in Figure 130. The two micrographs have almost the same grain size, indicating that the grain coarsening reaction results from exposure to temperature and is not strain induced.

6.2 ANALYSIS OF ISOTHERMAL COMPRESSION RESULTS

The true stress - true strain data for each strain rate/temperature combination of Appendix C was used to calculate various deformation parameters to aid in the interpretation of the stress - strain behaviour and help evaluate the metallurgical mechanisms occurring during isothermal compression. In particular, the strain rate sensitivity exponent of the Hollomon equation (m of equation 8a) and the activation energy (Q_a of equation 7) are of interest. This section outlines the analytical method used to calculate these parameters and presents the noteworthy trends.

6.2.1 Strain Rate Sensitivity Exponent

As outlined in section 2.3.3.1 the strain coinciding with the peak stress often corresponds to the onset of the metallurgical process that primarily controls the stress - strain behaviour. Therefore, it is useful to plot the peak stress against strain rate, Figure 131. Some interesting results from this plot are:

- a) For all temperature/strain rate combinations tested (except $1200^{\circ}\text{C}/10^{-1} \text{ s}^{-1}$) the 1250°C HIP'ed condition has a higher peak flow stress.
- b) The strain rate dependence of peak stress is greater at the higher test temperatures, i.e. the slope of the plots in Figure 131 is higher at higher temperatures. This implies that there is more variation in the mechanism controlling deformation as a function of strain rate at higher temperatures.
- c) Notwithstanding the previous point, the slope of all curves changes with strain rate, meaning that the strain rate sensitivity exponent varies with strain rate. The largest slope typically occurs at 10^{-3} to 10^{-4} s^{-1} and is larger at the higher test temperatures. This $\sigma_p - \dot{\epsilon}$ relationship concurs with the general characteristic $\sigma - \dot{\epsilon}$ curve for superplastic deformation shown in Figure 30a.

From the results of Figure 131 the strain rate exponent for each temperature/strain rate combination can be calculated. It should be noted that this calculation of the strain rate exponent (m) is for a strain level corresponding to the peak stress (σ_p) for each test condition. Therefore, the condition of equation 8b that m be calculated for a constant value of strain is not strictly obeyed. However, since for most test conditions σ_p occurs at a true strain close to 0.05, only a minimal error is introduced. The condition that m be calculated at a constant strain, is often interpreted to mean that m should be calculated for constant microstructure. Since, as previously stated, the onset of the metallurgical mechanism controlling deformation coincides with the peak stress, it can be assumed that up until σ_p the initial microstructure still exists. Thus, the calculation of m at σ_p corresponds to constant structure. For cases where the peak stress occurs at strain levels

much greater than 0.05 (e.g. $1200^{\circ}\text{C}/10^{-4} \text{ s}^{-1}$) the stress at a strain of 0.05 was substituted for the peak stress for the calculation of the strain rate exponent. Using this procedure the strain rate exponents calculated for each temperature/strain rate combination tested were used to determine contours of constant strain rate exponent by interpolation. Such constant strain rate contours are presented in Figure 132 as a function of test temperature and applied strain rate.

For both HIP'ed initial conditions the region of superplastic deformation ($m > 0.3$) is indicated in Figure 132. For both HIP'ed states, the strain rate exponent is strongly dependent on both test temperature and strain rate, varying from 0.1 to >0.4 for the 1050°C HIP'ed material and from 0.1 to ≈ 0.3 for the 1250°C HIP'ed material. In general, increasing temperature or decreasing strain rate results in a higher strain rate exponent. Deviation from this trend occurs in the strain rate range of 3×10^{-4} to $1 \times 10^{-4} \text{ s}^{-1}$ for the 1050°C HIP'ed material, where m decreases with decreasing strain rate.

To further analyse the effects of strain, the strain rate exponent for each temperature/strain rate combination was calculated at a true strain of 0.3 and 0.65. This former strain level typically represents a region of flow softening for the curve types 1 and 2, Figure 118. After 0.65 strain a constant or near constant flow stress has been attained.

The variation of the strain rate exponent as a function of strain is strongly dependent on the initial microstructure and the test conditions. As shown in Figure 133 for 1050°C HIP'ed material deformed at a strain rate of 10^{-1} s^{-1} , m remains constant as a function of strain for all test temperatures, except at 1200°C for which the strain rate exponent decreases with applied strain. In contrast, at a strain rate of 10^{-4} s^{-1} , Figure 133, the strain rate exponent as a function of strain depends on the test temperature. Most notable is the pronounced decrease in strain rate exponent with strain during compression at 1200°C , even though the flow hardening occurring is minimal, see Figure 118.

For the 1250°C HIP'ed initial condition, Figure 134 shows the m versus ϵ relationship. For a strain rate of 10^{-1} s^{-1} , m is approximately constant with strain for all test temperatures. This relationship corresponds to stress - strain curve types 1 and 2. At a strain rate of 10^{-4} s^{-1} the m either increases or decreases depending on the test temperature.

Note that for both HIP'ed conditions, curve types 1 and 2 of Figure 118, which exhibit marked flow softening, are characterized by an m value that remains constant or slightly increases with applied strain. In contrast for curve types 3 and 4, for which little or no flow softening occurs, the m value decreases with applied strain. These two types of behaviour are correlated with the microstructural changes occurring in section 6.3.2.

6.2.2 Activation Energy

The activation energy, Q_e , for isothermal compression is calculated from equation 7 presented in section 2.3.3.1. This formulation of activation energy includes the effect of stress level and test temperature, while strain effects are introduced by the inclusion of the strain rate sensitivity exponent, m .

The derivation of Q_e presented in Appendix A imposes the condition that Q_e be calculated for constant values of strain. However, in a similar manner to the calculation of m in the previous section, Q_e is calculated using the peak true stress value, which typically occurs near an applied true strain of 0.05. This approximation introduces a small numerical error into the activation energy calculations, but nevertheless useful information is acquired using this procedure. As before, in cases where the peak true stress occurs at strains much greater than 0.05, the stress level at a strain of 0.05 was used for calculating the activation energy.

To determine Q_e it is necessary to calculate $\frac{d \ln \sigma_p}{d(1/T)}$. This was done by plotting $\log \sigma_p$ against $1/T$ for both the 1050°C and 1250°C HIP'ed conditions as illustrated in

Figure 135. These plots show a linear relationship between σ_p and $1/T$ for each strain rate, with the slope $\frac{\Delta \ln \sigma_p}{\Delta 1/T}$ given for each strain rate in Figure 135. Combining this calculation with the appropriate m value from Figure 132 and the Universal Gas Constant ($R=8.31$ kJ/mole K) yields an activation energy for each temperature/strain rate combination tested.

Following the procedure of the previous three paragraphs, plotted in Figure 136 are interpolated contours of constant activation energy as a function of strain rate and temperature. At test temperatures of 1000°C and greater, the activation energy for deformation of the 1050°C HIP'ed condition is less than for the 1250°C HIP'ed condition. For example, for the test at 1000°C and 10^{-1} s⁻¹, the 1050°C HIP'ed material displays an activation energy of 300 kJ/mole compared to 372 kJ/mole for 1250°C HIP'ed material. At lower test temperatures the activation energies are similar. Note the range of activation energies in Figure 136 (160-570 kJ/mole) is similar to that reported for hot compression of other near γ -TiAl alloys (308-528 kJ/mole, see section 2.3.3.1). However, all of the previously reported activation energies were calculated for constant stress rather than constant strain, as is the case for the current analysis.

For 1050°C HIP'ed material with a curve type 1 stress - strain response, the activation energy is almost independent of the applied strain rate. For compression corresponding to the other curve types, the activation energy decreases with increasing temperature or increasing strain rate.

For material HIP'ed at 1250°C, the activation energy is only strongly dependent on the strain rate at low test temperatures and strain rates, i.e. the lower left quadrant of Figure 136b. At all other temperature/strain rate combinations the activation energy is approximately inversely related to temperature.

6.3 DISCUSSION OF ISOTHERMAL COMPRESSION

The purpose of this section is to appraise the results of the isothermal compression tests so that conclusions can be made regarding the hot pressing of TiAl+W. Two important aspects of the results and analysis are considered. Firstly, a pragmatic evaluation of the results is presented, which leads to conclusions regarding the forgeability of TiAl+W. Secondly, it is of interest to determine the deformation processes occurring and controlling hot compression behaviour throughout the temperature/strain rate range examined. As such, the microstructural results will be correlated with the stress - strain, strain rate sensitivity and activation energy results for each of the four generic responses to isothermal compression illustrated in Figure 118.

6.3.1 Forgeability of TiAl+W

Forging operations must maintain the soundness of the workpiece. Therefore, an important criterion for determining the forgeability is the tendency for cracks or voids to occur. Figures 113 to 116 and Table XVI summarize the cracking that develops during isothermal compression of TiAl+W. Using these results, a 'forging map', can be constructed indicating the temperature/strain rate regimes in which *good*, *suspect* or *poor* forgeability exists. For the TiAl+W alloy such forging maps are illustrated in Figure 137, in which it is implied that a lack of cracking after an applied true strain of ≈ 0.8 indicates acceptable forgeability. It is very important to note that for both initial HIP'ed conditions, temperature/strain rate combinations exist for which sound forgings are produced. The tensile results of the previous chapter indicated that the TiAl+W alloy does not exhibit a brittle-ductile transition at $\approx 700^\circ\text{C}$ (Table XV), as do many other near γ -TiAl alloys (Figure 13). A major question arising from the low ductilities of the tensile tests (4.5% maximum at 850°C) is whether the alloy is forgeable at any temperature/strain rate combination. The results of Figure 137 clearly illustrate that successful forging is possible under appropriate conditions. However, comparison of the *good* forgeability region of Figure 137 with that for Ti-48%Al-2%Cr-2%Nb (Figure 29) shows that TiAl+W requires

much higher temperatures and slower strain rates to produce a sound forging. This illustrates the dependence of forgeability to compositional factors. As indicated previously (see section 2.5.1), the addition of ternary chromium causes the formation of β , which is thought to improve the hot workability (see section 2.3.3). In contrast, in the current TiAl+W alloy, despite the presence of β much poorer workability is attained. It may be speculated that this difference in workability could be related to either, differences between chromium rich β and tungsten rich β , or effect of the ternary element in solid solution. As indicated in section 2.5.4, tungsten is known to impede diffusion assisted dislocation movement. This may reduce the ability of TiAl+W to accommodate strain during forging. Also, forgeability may be related to differences in the ductile-brittle transition of the two alloys.

From these forging maps, practical guidance can be obtained regarding the temperature/strain rate combinations to be used in production forging operations. Contours of peak true stress are superimposed on the forging maps of Figure 137. As expected, the peak true stress decreases with increasing temperature or decreasing strain rate. These constant stress contours are of importance to the forger, since they allow calculation of the approximate press force and power required for a particular operation. The force and power can be approximated from:

$$\begin{aligned} F &= \sigma_{tp} \cdot A_p \cdot Q \\ P &= \frac{\sigma_{tp} \cdot V \cdot \epsilon \cdot Q}{t} \end{aligned} \quad \dots \dots (28) ,$$

where:

F	is the press force,
σ_{tp}	is the peak true stress,
A_p	is the workpiece maximum plan area,
V	is the workpiece volume,
ϵ	is the total strain applied,
P	is the press power,
t	is the time for compression to ϵ ,
Q	is an interface pressure multiplying factor to account for friction ²⁰⁷ .

This formulation should yield conservative force and power requirements, since it assumes that the peak true stress value exists for all strain values. For example, Figure 137 indicates that for 1050°C HIP'ed material, good forgeability exists at 1200°C and 10^{-2} s^{-1} with a corresponding peak true stress of $\approx 130 \text{ MPa}$. If the workpiece volume is 0.01 m^3 (e.g. a preform of 30 cm diameter and 15 cm height), and it must be worked to a total true strain of 0.8 with a platen-workpiece friction coefficient of ≈ 0.2 (corresponding $Q \approx 1.2^{207}$) the corresponding force and power required are 11000 kN (≈ 1200 tons force) and 16.4 kW. Obviously, the forging press under consideration must be capable of operating at 1200°C and at a constant strain rate of 10^{-2} s^{-1} . Since most forging presses are rated by the maximum force that can be applied, this calculation provides a quick check of the ability of a given press to perform a specified operation. It is clear, then, that the information presented in Figure 137 provides the forging shop with useful knowledge.

6.3.2 Deformation Processes

In section 2.3.3.2 it was explained that superplastic deformation occurs when the strain rate sensitivity exponent is >0.30 . Hence, it is clear from Figure 132 that regions of non-superplastic and superplastic flow exist within the temperature/strain rate domain investigated. Given this change in behaviour, it is the purpose of this section to examine in more detail the deformation processes occurring throughout the temperature/strain rate ranges studied. Each of the stress - strain curve types introduced earlier in the chapter will be discussed individually. The temperature/strain rate combinations exhibiting each of these curve types is summarized in Table XVI.

6.3.2.1 Curve Type 1.

The predominant microstructural changes occurring during deformation with this type of stress - strain relationship is strain hardening by deformation twinning and increasing dislocation density (Figure 124). This corresponds to the highest activation energy, (always $>400 \text{ kJ/mole}$) which decreases with increasing temperature since

dislocations become more thermally activated. The small drop in flow stress at true strains greater than ≈ 0.30 (Figure 118) is due to a combination of some dynamic recrystallization occurring and adiabatic heating. The former effect is evidenced by small dislocation free grains, which nucleate at the intersection of deformation twins and grain boundaries, Figure 124, similar to previous observations²⁰⁸. For an m value of 0.15 the power partitioning index η (equation 9) is only 0.26, indicating that about one quarter of the forging work applied is required to cause the microstructural changes occurring. The remainder of the work applied is used to overcome friction effects and cause adiabatic heating of the sample. Since the platen-sample coefficient of friction is estimated to be < 0.04 (see section 3.8), a significant portion of the work causes adiabatic heating. At the fast strain rate of 10^{-1} s^{-1} , such adiabatic heating causes softening with applied strain, since there is little time for the adiabatic heat created to be conducted into the slightly cooler platens.

6.3.2.2 Curve Type 2.

Consistent with the isothermal forging behaviour outlined in section 2.3.3.1, dynamic recrystallization occurs during compression according to curve type 2, Figure 123a, being initiated at the peak stress. The ease with which dynamic recrystallization occurs in TiAl is due to the low mobility of dislocations²⁰⁹. As outlined in section 2.2 the high Peierls stress and superdislocation dissociation limits slip and recovery processes. As a result, a dislocation density sufficient to raise the internal strain energy above that required to activate dynamic recrystallization is achieved. It is evident from Figure 123a that dynamic recrystallization causes refinement of the coarse grains (e.g. regions surrounding prior dendrites) and coarsening of the fine grains. Thus, as observed in other near γ -TiAl alloys^{103,209}, a dynamic equilibrium occurs between grain refining and grain coarsening, and this serves to improve the microstructural homogeneity. The increasing volume fraction of recrystallized grains with strain causes flow softening and lowers the dislocation density. Thus, at a true strain of 0.8 an almost completely

recrystallized structure with a low dislocation density exists, Figure 126, and the relatively strain rate exponent (m) at $\epsilon=0.8$ between 0.16 and 0.25.

At true strains greater than ≈ 0.4 a nearly uniform flow stress is achieved, indicating that microstructural dynamic equilibrium exists. Accordingly, there is a balance between dislocation generation, caused by increasing strain, and dislocation annihilation and grain growth, due to the elevated temperature, and dynamic recrystallization causing grain refinement. Such a microstructurally stable condition should lead to good forging properties. Unfortunately however, at the temperature/strain rate combinations compatible with curve 2 type forging response, cracks develop after an applied true strain of only 0.05 (Figure 116), before the dynamic recrystallization process is initiated.

The activation energy corresponding to curve type 2 deformation ranges from ≈ 200 to ≈ 420 kJ/mole, Figure 136. This range encompasses the values of activation energy reported in the literature for hot compression of near γ -TiAl alloys during which dynamic recrystallization and flow softening was observed^{86,91,208} and is less than that for curve type 1. It is expected that the driving force for dynamic recrystallization is the increase in internal strain caused by deformation twinning and the generation of dislocations. This is similar to the deformation mechanisms for curve type 1, however due to the higher temperatures (greater thermal activation) and/or slower strain rates (which allows a longer time for initiation of dynamic recrystallization at any instantaneous strain) the internal strain level required to initiate dynamic recrystallization is lower. This results in a lower peak stress and hence activation energy compared to curve type 1.

Within the temperature/strain rate domain resulting in curve type 2 forging response (Figure 136) the activation energy is approximately inversely related to both temperature and strain rate. The temperature dependence is due to increased thermal activation. With increasing strain rate, it would be expected that development of a dislocation substructure would be more rapid, thus providing a larger driving force for dynamic recrystallization and thus a lower peak stress. However, the faster strain rates,

cause higher peak stresses - giving the higher activation energy - because the initiation of dynamic recrystallization is not fast enough to balance the generation of new dislocations caused by the faster strain rate. However, once dynamic recrystallization is initiated, it proceeds at a sufficiently fast rate to lower the dislocation density, thus causing softening. Hence, at lower temperatures and faster strain rates a more pronounced peak stress occurs.

The strain rate sensitivity exponent remains nearly constant with applied strain during curve type 2 deformation, with m values in the range 0.16 to 0.25. This indicates that the mechanism controlling deformation is not changing appreciably with strain beyond the peak stress. This is consistent with the existence of microstructural dynamic equilibrium that maintains a constant microstructure. An interesting exception to this situation occurs for 1250°C HIP'ed material deformed at 10^{-4} s^{-1} (Figure 134b), for which the m value increases with strain. The 1250°C HIP'ed condition has a coarser initial microstructure (Figure 78), resulting in lower peak stress m values. However, the dynamic recrystallization may cause substantial grain refinement giving improved forgeability and a corresponding higher strain rate exponent.

6.3.2.3 Curve Type 3.

Similar to curve type 2, a peak in the true stress is still observed in curve type 3 deformation corresponding to the onset of dynamic recrystallization. However, an important difference from the curve type 2 behaviour, is that $m > 0.3$. Hence, superplastic flow occurs and grain boundary sliding will be a significant deformation mode. Due to the concurrent operation of dynamic recrystallization and grain boundary sliding a steady state flow stress is achieved at a relatively low strain, ≈ 0.15 . However, with increasing strain, deformation by grain boundary sliding is reduced as evidenced by the decreasing m value, Figure 133a. Thus, to maintain a steady state flow stress, the volume fraction of dynamically recrystallized grains must increase with strain.

It is interesting that the activation energies of ~ 200 kJ/mole typical for this curve type agree well with a recently reported activation energy for grain boundary diffusion controlled (Coble) creep (Q of equation 14) in a near γ -TiAl alloy of 192 kJ/mole ¹⁴⁶. The accommodation of grain boundary diffusion by grain boundary sliding is a characteristic of both grain boundary creep ¹⁴³, and superplasticity. It is suggested, then, that the activation energy of ~ 200 kJ/mole corresponds to this deformation mechanism.

The activation energy (based on the peak stress), for curve type 3 compression decreases at higher temperatures and strain rates. The temperature effect is due to increased grain boundary sliding. At faster strain rates, substructure development will be more rapid, increasing the driving force for dynamic recrystallization, thus lowering the peak stress.

6.3.2.4 Curve Type 4.

For this type of compression behaviour, superplastic flow is a major deformation process, with m at the peak stress as high as 0.44. In contrast to the type 3 stress - strain behaviour, dynamic recrystallization does not occur, since minimal substructure development occurs. The absence of dynamic recrystallization is evidenced by the continued presence of localized areas of large grains (up to 40 μm) surrounding the prior dendritic regions, as seen in the HIP'ed condition, Figure 123b. This differs from curve type 2 compression, where dynamic recrystallization occurs, and the prior dendrites are surrounded by finer grains, Figure 123a. Thus, for curve type 4 the occurrence of superplasticity depends only on the initial as-HIP'ed grain size, rather than being associated with grain size modification by dynamic recrystallization.

As mentioned in the previous section, the activation energy for superplasticity and grain boundary sliding are thought to be similar. However, for the superplasticity occurring during curve type 4 compression the activation energy is substantially higher than the 200 kJ/mole presented in the previous section. This suggests that the rate

controlling process is something other than grain boundary sliding. To accommodate grain boundary sliding, grain shape changes are necessary if cracking is to be avoided. These shape changes occur by dislocation or diffusional processes ¹⁰¹. The activation energies observed of ≈ 350 kJ/mole, are consistent with those reported for creep controlled by diffusion via dislocation pipes (300–410 kJ/mole, section 2.4.3) and so it may be speculated that these deformation mechanisms produce the grain shape changes necessary and are the rate controlling process. This in agreement with some models for superplasticity which suggest pipe diffusion as the controlling process ²¹⁰.

The high temperature and slow strain rates consistent with curve type 4 deformation results in considerable grain coarsening, as seen in Figure 123b. The grain coarsening reaction causes a substantial drop in the strain rate exponent, Figure 138, eliminating the superplastic flow at strains slightly higher than the strain for peak stress. Once the m value drops below 0.30 strain accommodation will occur by development of the dislocation substructure evident in Figure 123. However, at the high temperatures and slow strain rates involved, it is postulated that recovery processes and dislocation migration to grain boundaries will prevent an extensive substructure from forming.

6.3.3 Deformation Map

From the discussion in the preceding sections, it should be apparent that for each of the four curve types a different deformation mechanism is predominant. By combining these microstructural results, the analytical results of section 6.2 (calculation of m and activation energy) with the stress - strain curves of Figure 118 and Appendix C, the temperature/strain rate regimes in which each deformation mechanism is predominant can be identified. The 'deformation maps' generated from this analysis are illustrated in Figure 139. Comparison with the forging map of Figure 137, indicates that to produce crack free forgings invariably requires compression within the temperature/strain rate domain compatible with superplastic flow. This means that accommodation of the deformation occurs predominantly by grain boundary sliding and/or motion of grain

boundary dislocations. In this manner, grain boundaries act as channels along which plastic flow occurs. With increasing grain size, the grain boundary area decreases and plastic flow can no longer be absorbed by the grain boundaries, forcing plasticity to intragranular regions. Since these regions have a very low plastic capacity, cracking and poor forgeability occurs. Accordingly, successful forging requires a fine grain size, as well as a temperature/strain rate combination suitable for superplasticity, implying $m > 0.3$.

In region 3 of the deformation map, Figure 139, the fine grain size required for superplastic flow is maintained by microstructural dynamic equilibrium caused by dynamic recrystallization. Interestingly however, in region 4, grain coarsening occurs eliminating superplastic flow at strains just beyond the strain required for the peak stress, Figure 138. Nevertheless, crack free forgings are still produced. The grain coarsening reaction requires substantial grain boundary motion and it is postulated that the migrating grain boundaries absorb the intragranular dislocations generated as a result of the compressive strain, thus relieving the internal strain and allowing crack free forgings to be produced.

7. GENERAL DISCUSSION

A primary objective of this thesis work was to explore the HIP plus forge processing route for powder metallurgically produced near γ -TiAl alloys. The results discussed in the preceding three chapters indicate that engineering strains of $\sim 120\%$ can be applied to produce sound forgings, if the HIP'ed microstructure and the forging conditions (temperature and strain rate) are compatible with superplastic flow. This is an important result, verifying that complex shapes could be produced via this processing route. In this chapter, several important aspects of the results are discussed with regard to their influence on fabrication and service. From this discussion, issues requiring further research work become apparent.

It is evident from the results, that the HIP'ed microstructures can deform superplastically during forging. Indeed, as discussed in the previous chapter, superplastic deformation is a prerequisite for successful forging. The deformation maps of Figure 139 show that the temperature/strain rate window in which superplastic flow occurs is isolated to high temperatures and slow strain rates. From a manufacturing viewpoint, it would be desirable to enlarge the temperature/strain rate window at which superplastic flow occurs. For this to happen necessitates modification to the microstructure of the forging preforms. A microstructure optimized for superplasticity, would probably contain a uniform fine grain size. Although, the HIP'ed microstructures described in chapter 5, do have a fine grain size, they do not have a uniform microstructure. Thus, it is of interest to consider modifications to the processing route to improve microstructural homogeneity.

The major feature contributing to microstructural inhomogeneity is the presence of well defined prior dendritic and interdendritic regions in the HIP compacts. These two well defined areas could be clearly identified in the gas atomized powder and stem from the microstructural evolution occurring during solidification and cooling of the powder particles. Thus, it is possible that the powder microstructures could be altered by changing the thermal conditions existing during gas atomization. Although discussion of this aspect of the processing is beyond the scope of this thesis, the gas atomization parameters are primarily determined by the requirement to produce spherical powder of a uniform size distribution. Given this situation, it is considered unlikely, that an opportunity exists for the development of improved microstructural uniformity by modification of the gas atomization process parameters. Therefore, it is necessary to improve the microstructural homogeneity by thermomechanical processing prior to forging.

The major processing step prior to forging is HIP'ing. Therefore, methods of improving microstructural homogeneity during HIP'ing should be considered. The HIP temperatures studied in this thesis (1025°C to 1250°C), correspond to the $\alpha_2+\gamma$ and $\alpha+\gamma$ phase fields of the phase diagram (Figure 12). This temperature range was selected for study based on the requirement to produce full density during HIP'ing (see section 2.3.2.3). Within this temperature range two important trends were observed. Increasing HIP temperature resulted in improved homogeneity but also a larger grain size. While the former trend is thought to be desirable for forgeability, the latter is not. Superimposed on these trends are significant differences in the volume fraction and morphology of the α_2 , γ and β phases, which may also influence forging response. Thus, the HIP temperature required to produce a microstructure optimized for forgeability is not immediately apparent.

The effect of heat treatment in the $\alpha+\gamma$ and α phase fields is well established (see section 2.3.4). Moreover, it has been demonstrated that in the current TiAl+W alloy heat treatment in the α phase field results in a uniform fully transformed structure, similar to Figure 8d²¹⁰. Unfortunately, the grain growth kinetics during such heat treatments are rapid, making it difficult to produce a fine grained fully transformed microstructure.

However, the possibility exists that HIP treatments in the α phase field ($>1400^{\circ}\text{C}$), could result in this microstructure. During such HIP'ing the temperature would be sufficient to develop a fully transformed - and thus uniform - microstructure. Concurrently, the deformation required for consolidation could result in dynamic recrystallization, giving a fine grained fully transformed microstructure. It is already apparent from this thesis work that dynamic recrystallization can occur due to the deformation associated with HIP consolidation. For such a fine grained fully transformed microstructure to be developed during HIP'ing, would require 'balancing' of the transformation, grain growth and dynamic recrystallization kinetics. Currently, insufficient knowledge exists to definitively define appropriate HIP cycles. However, it is known that in the single phase α field the transformation and grain growth kinetics are very rapid ²¹. Therefore, the HIP time necessary at the single phase α temperature would be relatively short.

An important aspect of this discussion is that the HIP cycle is no longer considered to be a process solely aimed at developing full density. Instead, HIP'ing becomes an important thermomechanical processing step that is optimized to produce a microstructure ideally suited for the succeeding processing steps.

Alternately, the HIP'ed cycle could be optimized to produce a microstructure suitable for direct service applications. Such a microstructure would require mechanical properties compatible with the intended application (ductility, creep, fatigue, etc.). For this purpose, rather than the fine grained HIP'ed microstructure, a coarser grain size would probably be desirable. Whether a fully lamellar microstructure would be optimum is not readily clear from the available literature on near γ -TiAl alloys. However, once again, the HIP cycle would have the dual purpose of full densification of the powder and appropriate control of the microstructure to produce the needed mechanical properties.

This thesis work has concentrated on determining the suitability of the HIP plus forge processing route for near γ -TiAl alloys. The forged microstructures were examined with the objective of understanding the deformation processes active during compression.

However, application of forged parts will probably require modification of the deformed microstructure for the development of suitable mechanical properties. The post-forge heat treatments required for this purpose have not been studied as part of this thesis work. The main reason for this situation is that considerable further work is required to optimize the response of near γ -TiAl alloys to processing. Once the processing parameters have been further optimized, development of final heat treatments can be applied to material processed using these optimized parameters.

8. SUMMARY & CONCLUSIONS

- 1. A comprehensive review of near γ -TiAl technical knowledge has been completed. From the information contained in this review a powder metallurgically produced alloy of TiAl+W was selected for this thesis research.**
- 2. The microstructural evolution during solidification and cooling of both Ti-54%Al and Ti-48%Al-2%W has been determined. In the Ti-54%Al powder α dendrites form, while β dendrites form in the TiAl+W alloy. In the ternary alloy the β dendrites transform to martensitic α and are surrounded by peritectic α resulting from the $\beta+L \rightarrow \alpha$ reaction. In the ternary powder, formation of the β dendrites causes aluminum enrichment of the remaining liquid, resulting in secondary nucleation of α . These secondary dendrites are then surrounded by γ .**
- 3. For both Ti-54%Al and TiAl+W alloys, full densification is achieved by HIP'ing at 1100°C and 200 MPa for 2 hours.**
- 4. The HIP consolidated microstructure of TiAl+W consists of well defined prior dendritic and interdendritic regions. With increasing HIP temperature the volume fraction of dynamically recrystallized grains and the grain size of these two regions increases. The dynamic recrystallization improves the microstructural homogeneity. Deformation during HIP'ing is primarily accommodated by the interdendritic areas.**

5. **During HIP'ing third phase tungsten rich body centred cubic β particles develop. The kinetics of the β formation and dynamic recrystallization are very dependent on the HIP temperature.**
6. **The different microstructures of the prior dendritic and interdendritic regions in the 1050°C and 1100°C HIP'ed conditions, cause non-uniform tensile deformation and poor ductility at room temperature and 550°C. The improved ductility of the 1250°C HIP'ed condition is due to the more uniform microstructure and consequent greater homogeneity of deformation.**
7. **The 850°C tensile deformed structure of all HIP'ed conditions is more homogeneous, resulting in improved ductility.**
8. **For the TiAl+W, the tensile strength at room temperature and 550°C displays a Hall-Petch effect. The best combination of ambient temperature strength and ductility is 505 MPa and 1.3 %, obtained in 1250°C HIP'ed material. At 850°C tensile failure occurs intergranularly and along prior particle boundaries, thus eliminating the Hall-Petch effect.**
9. **Isothermal compression in the temperature range 900°C to 1200°C further improves the microstructural homogeneity. However, prior dendritic regions are still evident.**
10. **For the TiAl+W alloy, the true stress - true strain, strain rate sensitivity exponent, activation energy and microstructural changes occurring during isothermal compression are dependent on the applied temperature and strain rate. Four distinct deformation processes can be identified allowing construction of a 'deformation map', which relates the deformation process occurring during compression with the temperature and strain rate applied. With increasing temperature and decreasing strain rate the predominant deformation mechanism**

evolves from twinning and dislocation substructure development, through dynamic recrystallization, to combined dynamic recrystallization and superplasticity and finally superplasticity and grain coarsening.

- 11. Sound forgings after 120% engineering strain can be produced from TiAl+W. However, for sound forgings the temperature/strain rate combination must be such that superplastic flow ($m > 0.3$) is a significant deformation mechanism.**
- 12. The temperature/strain rate domain for which superplastic flow occurs is larger for 1050°C HIP'ed material than 1250°C material, due to the smaller initial grain size of the 1050°C HIP'ed material ($\approx 5 \mu\text{m}$ versus $13 \mu\text{m}$).**
- 13. Superplastic deformation during forging can be maintained to higher strains when the temperature and strain rate applied also causes concurrent dynamic recrystallization. At these temperature/strain rate combinations a microstructural dynamic equilibrium exists, with dynamic recrystallization maintaining the fine grain size required for superplastic flow, as well as accommodating the grain shape changes necessary for superplasticity.**

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TABLE I

**PROPERTIES OF TITANIUM ALUMINIDES, TITANIUM ALLOYS
AND SUPERALLOYS**

Ref. 2

PROPERTY	Ti-BASE ALLOYS	α_2 -Ti ₃ Al ALLOYS	γ -TiAl ALLOYS	SUPER- ALLOYS
Density	4.5	4.1-4.7	3.7-3.9	8.3
Room Temp. Modulus	96-115	120-145	160-176	206
Y.S. (MPa)	380-1150	700-990	400-630	-
U.T.S. (MPa)	480-1200	800-1140	450-700	-
Creep Limit (°C)	600	750	1000	1090
Oxidation Limit (°C)	600	650	850	1090
Room Temp. Ductility	10-20	2-7	1-3	3-5
High Temp. Ductility	high	10-20	10-20	1020
Structure	hcp/bcc	DO ₁₉	L1 ₀	fcc/L1 ₂

TABLE II

PHASES PRESENT IN TiAl POWDER

Ref. 31

POWDER MICROSTRUCTURE	POWDER PARTICLE SIZE RANGE			
	<44 μ m	44-63 μ m	83-88 μ m	88-150 μ m
Single phase α_2	30%	13%	3-5%	<1%
Dual phase $\alpha_2+\gamma$	69%	85%	93%	95%
Single phase γ	<1%	1-2%	2-4%	3-5%

TABLE III
 γ -TiAl HIP CYCLES FOR FULL CONSOLIDATION

POWDER TYPE	POWDER SIZE (μm)	COMPOSITION	HIP CYCLE FOR FULL DENSITY	HIP'ED GRAIN SIZE (μm)	REF
PREP	130 avg	γ -Ti-48%Al	982°C, 207 MPa	9-34	68
TGA	<500	Ti-48Al-1V	1000°C, 200 MPa, 4 hours	10-20	73
Cent. Atom.	80-250	Ti-50Al	1200°C, 98 MPa, 3 hours	~30	84
Cent. Atom.	80-250	Ti-47Al-2V	1200°C, 98 MPa, 3 hours	~10	84
TGA	100-250	Ti-32wtAl-1wtV	1100°C, 140 MPa,		81
TGA	100-250	Ti-36wtAl-5wtNb	1100°C, 140-275 MPa	~3	81
RSR	<300	Ti-48Al-2.4Nb-.3Ta	1066°C, 182 MPa, 4 hours	1.2 (γ) 20 ($\alpha_2+\gamma$)	79
REP	60-300	Ti-50Al-1.8Nb	1066°C, 182 MPa, 4 hours	28	18

PREP - Plasma Rotating Electrode Process
 TGA - Titanium Gas Atomization
 Cent. Atom. - Centrifugally Atomized
 RSR - Rapid Solidification Rate Process (centrifugal atomization)
 REP - Rotating Electrode Process

TABLE IV
TENSILE PROPERTIES OF Ti-48Al-3(Cr+Mn)-2Nb
POWDER ALLOY EXTRUSION

Ref. 24

HEAT TREATMENT	MICROSTRUCTURE	Y.S. MPa	U.T.S. MPa	%EL.
1230°C/4 hr.	fine equiaxed γ	494	531	1.6
1275°C/4 hr.	Fig. 8b,	471	535	1.8
1325°C/2 hr.	Fig. 8c,	477	542	1.9
1350°C/2 hr.	large grain lamellar	-	440 *	0.4
1350°C/½ hr.	Fig. 8d,	-	431 *	0.4

All heat treatments followed by cooling at 80-100°C/hr and 940°C/6 hr and air cooled.

* Fracture strength given, sample failed before 0.2% yield.

TABLE V
EFFECT OF MICROSTRUCTURAL CONDITION ON
FRACTURE TOUGHNESS OF Ti-48Al-2Nb-2Cr

Ref. 6

MICROSTRUCTURE	FRACTURE TOUGHNESS MPa√m
equiaxed γ	18.8
equiaxed γ , some lamellar	26.7
50% equiaxed γ , 50% lamellar	31.3
fully lamellar	32.5

TABLE VI
TENSILE PROPERTIES OF Ti-48Al-1V

HIP'ed 1000°C/200 MPa/4 hr.
Heat Treated 1260°C/4 hr AC + 1093°C/8 hr AC

Ref. 73

TEMPERATURE	U.T.S. (MPa)	Y.S. (MPa)	%ELONG.	% RED. OF AREA
RT	466	420	1.2	3.2
649	395	358	1.0	3.6
760	454	344	4.0	6.0
871	386	282	46.0	35.3

TABLE VII
MICROSTRUCTURAL EFFECTS IN γ BASED ALLOYS

Ref. 44

PROPERTY	CONDITIONS	BENEFICIAL STRUCTURAL FEATURES	OPTIMAL MICRO- STRUCTURE
Strength	<760°C	Small Grains Lamellar Structure	Duplex
Ductility	<760°C	γ/α ratio	Duplex
Toughness	All temperatures	Lamellar Structure	Fully Lamellar
Creep & Rupture	<650°C	Small Grains Lamellar Structure	Duplex
	>650°C	Large Grains Lamellar Structure	Fully Lamellar
High Cycle Fatigue	>250 MPa, 760°C	Small Grains Lamellar Structure	Duplex
	<250 MPa, 760°C	Large Grains Lamellar Structure	Fully Lamellar

TABLE VIII
EFFECT OF TUNGSTEN ON TENSILE PROPERTIES

Ref. 117, 152, 162

COMPOSITION & TEMPERATURE (°C)	Y.S. MPa	U.T.S. MPa	%Elong.	%R.A.
Ti-50%Al				
25	335	450	0.9	1.6
700	300	360	14.0	22.0
Ti-49%Al-2%W				
25	599	736	0.7	1.3
260	523	692	0.9	1.9
500	582	828	1.5	3.9
700	528	800	3.2	4.9
800	488	732	5.1	8.5
900	385	504	2.3	8.0

* Values listed for U.T.S. are actually fracture stresses.

All material produced from PREP powder, extruded 26:1 reduction at 1400-1415°C. Tested in as extruded condition.

TABLE IX
TIME TO 1% CREEP STRAIN (IN HOURS) FOR Ti-48Al-2.2W

REF. 152, 162

CREEP LOAD & TEMPERATURE MPa & °C	AS EXTRUDED	HEAT TREATMENT 'A'	HEAT TREATMENT 'B'
386 & 700	273	197	127
276 & 760	260	130	25
172 & 850	96	26.8	-

All material compacted from PREP powder by extrusion at 1400-1415°C with 26:1 ratio.

HEAT TREATMENT 'A' - 1150°C/2 hr FC + 950°C/8 hr FC (under vacuum).

HEAT TREATMENT 'B' - 800°C/2 hr FC + 950°C/8 hr FC (under vacuum).

TABLE X
PROPERTIES OF AS EXTRUDED Ti-45%Al+7.5vol%TiB₂

Ref. 184

CONDITION	Y.S. (MPa)	U.T.S. (MPa)	%Elong.
As-extruded	675	760	0.7
As-extruded+10hr/1150°C	705	800	0.8
As-extruded+10hr/1200°C	675	775	0.7
As-extruded+10hr/1250°C	650	855	0.7
As-extruded+16hr/1200°C+10hr/900°C	675	850	1.1
As-extruded+16hr/1200°C+10hr/1000°C	650	760	0.9
As-extruded+16hr/1200°C+10hr/1100°C	660	770	0.95

TABLE XI
CHEMICAL COMPOSITION OF POWDER SAMPLES

Ref. 192, 193

ELEMENT	WEIGHT PERCENT	
	Ti-54Al	Ti-48Al-2W
Al	38.9 (53.1at%)	32.5 (48.4at%)
W	-	8.6 (1.9at%)
Fe	0.010	0.05
C	0.005	0.010
N	0.004	0.004
O	0.068	0.083
H	-	0.0016
Ti	bal.	bal.

TABLE XII

$h\mathbf{g}$ FOR TEM BEAM OPERATION NEAR $[110]$

BURGERS VECTOR	$g-1\bar{1}1$	$g-\bar{1}11$	$g-002$
$\frac{1}{2}[110]$	0	0	0
$\frac{1}{2}[1\bar{1}0]$	1	-1	0
$[101]$	2	0	2
$[\bar{1}01]$	0	2	2
$[011]$	0	2	2
$[0\bar{1}1]$	2	0	2
$\frac{1}{2}[112]$	1	1	2
$\frac{1}{2}[\bar{1}12]$	0	2	2
$\frac{1}{2}[1\bar{1}2]$	2	0	2
$\frac{1}{2}[\bar{1}\bar{1}2]$	1	1	2

TABLE XIII

COMPOSITIONAL ANALYSIS OF HIP'ED Ti-48%Al-2%W

ELEMENT	COMPOSITION (wt%)	
	1150°C HIP	1250°C HIP
W	8.7	9.3
C	0.011	0.0108
N	0.0049	0.0022
O	0.073	0.079
Fe	0.028	0.029

TABLE XIV
ROOM TEMPERATURE TENSILE RESULTS

ALLOY	HIP TEMP. (°C)	0.2% Y.S. (MPa)	FRACTURE STRESS (MPa)	% El.	E (GPa)
Ti-54%Al	1025*	-	256	0.1	-
Ti-48%Al-2%W	1050	619	637	0.9	151
	1100	-	588	0.6	145
	1100	605	607	0.6	158
	1150	-	494	0.3	226
	1150*	-	513	0.3	180
	1250	486	499	1.0	151
	1250	488	510	1.5	194

* Failed outside gauge length, fracture stress is maximum stress applied to gauge length.

TABLE XV
ELEVATED TEMPERATURE TENSILE RESULTS Ti-48%Al-2%W

TEST TEMP. (°C)	HIP TEMP. (°C)	Y.S. (MPa)	U.T.S. (MPa)	FRACTURE STRENGTH (MPa)	% EL.
550	1100	522	-	553	0.8
	1150	492	-	505	0.8
	1150	-	-	500	0.4
	1250	410	-	466	1.5
	1250	411	-	430	0.9
850	1100	324	388	377	4.5
	1100	325	384	379	4.7
	1150	354	-	403	2.3
	1150	353	-	402	1.8
	1250	348	409	405	4.5
	1250	337	398	396	4.2

TABLE XVI

**INCIDENCE OF CRACKING DURING ISOTHERMAL COMPRESSION &
STRESS - STRAIN CURVE TYPE**

HIP TEMP & STRAIN RATE (s ⁻¹)	TEST TEMPERATURE (°C)			
	900	1000	1100	1200
1050°C HIP				
10 ⁻¹	E 1*	EP 2	E 2	E 3
10 ⁻²	E 1	E 2	E 2	S 3
10 ⁻³	E 1	E 2	C 3	S 4
3x10 ⁻⁴	N/T	E 3	S 4	S 4
10 ⁻⁴	E 2	E 2	S 4	S 4
1250°C HIP				
10 ⁻¹	E 1	E 2	E 2	S 2
10 ⁻²	E 1	S 2	C 2	C 2
10 ⁻³	E 2	S 2	E 2	S 2
10 ⁻⁴	E 2	S 2	S 4	N/T

E Edge radial cracks,
C Centre line cracks,
P PPB cracks,
S Sound (no cracks),
N/T Not tested,

* Numbers classify
shape of σ - ϵ curve
according to Figure 118.
For details see
section 6.2 & Appendix C.

Figure 1. Schematic of gas turbine engine illustrating some potential applications of titanium aluminides. (Ref. 3)

Figure 2. Schematic of ordered face centred slightly tetragonal L1₀ structure of γ-TiAl, with Burgers vectors of relevant dislocations shown. Aluminum atoms (open circles) are located on (002) planes. (Ref. 13)

Figure 3. Proposed phase diagram for Ti-Al system, showing equilibrium and rapidly quenched phases. (Ref. 13)

Figure 4. Specific volume of unit cell for quenched TiAl as a function of aluminum content. (Ref. 16)

Figure 7. The stacking sequence and atomic arrangements in dual phase near γ -TiAl. Subscripts: M-matrix, SF-stacking fault and T-twin. (Ref. 22)

Figure 8. Microstructure of Ti-48.2%Al-3%(Cr+Mn)-2%Nb powder alloy after extrusion and solution treatments:
a) 1130°C/4 hr.
b) 1275°C/4 hr.
c) 1325°C/4 hr.
d) 1350°C/4 hr.
All treatments followed by cooling at 70-100°C/min. and 940°C/6 hr.
(Ref. 24)

Figure 9. Comparison of two proposed phase diagrams for the Ti-Al system. Dotted lines show the phase boundaries indicated in Figure 3, while the solid lines from reference 29, indicate the presence of the α phase formed by a peritectic reaction. (Ref. 29)

Figure 10. Partial Ti-Al phase diagram based on optical metallographic results. (Ref. 29)

Figure 11. Modified partial Ti-Al phase diagram consistent with experimental observations. (Ref. 33)

Figure 12. Complete Ti-Al phase diagram reflecting modified high temperature phase fields in the $\approx 50\%$ Al region. (Ref. 13)

Figure 13. The effect on strain of the brittle-ductile transition for Ti-52%Al. (Ref. 37)

Figure 14 Hall-Petch plot of room temperature yield stress of near γ -TiAl as a function of average grain diameter. (Ref. 43.)

Figure 15. Map of thermodynamic phase selection hierarchy. Solid lines are curves for solidification of α , β and γ phases and dashed lines are for solid state transformations between α , β and γ . (Ref. 34)

Figure 16. Schematic diagram of 'titanium gas atomization' process of Crucible Compaction Metals. (Ref. 69)

Figure 17. Typical particle size distribution for gas atomized γ -TiAl. (Ref. 69)

Figure 18. HIP cycle and densification history for Ti-45%Al. (Ref. 83)

Figure 19. Consolidation curves for Ti-50.3%Al-5%Nb, showing influence of temperature and time on HIP densification. (Ref. 80)

Figure 20. Effect of HIP time and temperature on pore density of Ti-48%Al-1%V at a) interior and b) edge of cross-section. (Ref. 81)

Figure 21. Experimental and predicted consolidation curves (for 182 MPa HIP pressure) for alloy of Figure 19. Dominant densification mechanism plotted; PL-CRP1 - power law creep, stage 1; PL-CRP2 - power law creep, stage 2; NH-CRP1 - Nabarro-Herring creep, stage 1. (Ref. 80)

Figure 22. Variation of grain size with HIP temperature for Ti-50%Al-4%Nb. (Ref. 81)

Figure 23. Comparison of grain size distributions in Ti-50%Al-4%Nb for HIP'ing pressures of a) 140 MPa, b) 210 MPa, c) 275 MPa. (Ref. 81)

Figure 24. True stress - true strain curve for cast and PM processed Ti-50%Al. (Ref. 86)

Figure 25. Relationship between true stress and Zener-Hollomon parameter for near γ -TiAl, $Q=308$ kJ/mole.

Figure 26. Relationship between deformed dynamically recrystallized grain size and peak stress for Ti-50%Al. (Ref. 86)

Figure 27. Relationship between grain size after dynamic recrystallization and peak true stress for Ti-50%Al. (Ref. 86)

Figure 28. Relationship between peak flow stress and Zener-Hollomon parameter during isothermal forging of TiAl+V. (Ref. 96)

Figure 29. Deformation map for Ti-48%Al-2%Cr-2%Nb. Dashed line shows boundary between poor and good forgability regions after 80% true strain. (Ref. 91)

Figure 30. The relationship between (a) stress and strain rate and (b) strain rate sensitivity index m for superplastic deformation. (Ref. 102)

Figure 31. Heat treatment schemes for titanium aluminides. (Ref. 108,109)

Figure 32. Lamellar volume fraction (f_v) and γ grain size for Ti-48%Al produced from powder as a function of heat treatment temperature and hold time. (Ref. 110)

Figure 33. Tensile properties as a function of temperature for Ti-48%, 52% and 54%Al binary alloys produced by melt spinning. (Ref. 115)

Figure 34. Effect of $\alpha_2+\gamma$ orientation on crack growth resistance. (Ref. 130)

Figure 35. Density normalized rupture life for various high temperature alloys. (Ref. 137)

Figure 36 Creep behaviour of near γ -TiAl alloy at 900°C in vacuum. (Ref. 138)

Figure 37. Stress dependence of the minimum creep rates in compression for Ti 53.4%Al, for grain sizes from 25-70 μm . (Ref. 147)

Figure 38. Power law creep behaviour of cast Ti-48%Al-2%Nb-2%Cr. (Ref. 150)

Figure 39 Fatigue crack growth rates for Ti-48%Al at two temperatures and stress ratios, compared to Ti-6%Al-4%V. (Ref. 161)

Figure 40. Effect of HIP temperature on ultimate tensile strength of Ti-50%Al-4%Nb. (Ref. 81)

Figure 41. Variation of compressive yield strength with temperature for Ti-50.9%Al. (Ref. 84)

Figure 42. Creep properties for various near γ -TiAl alloys, expressed as Larson Miller parameter (L-M).

Figure 43. The effect of ternary elements on the lattice dimensions of TiAl. (Ref. 46)

Figure 44. Effect of Cr additions on the mechanical properties of TiAl, when substituted for aluminum (open circles) or titanium (closed circles). (Ref. 163)

Figure 45. Room temperature stress-strain curves for (A) binary TiAl and (B) TiAl+2wt% Mn. (Ref. 169)

Figure 46. Creep strain rate for PM alloys, extruded at 1410°C in the as-extruded condition and after heat treatment, 2 hr/1150°C+8 hr/950°C (Ref. 145)

Figure 47 Comparison of weight change during oxidation of binary and TiAl+W alloys. (Ref. 174)

Figure 48. Effect of Nb additions on mechanical properties of TiAl, when substituted for aluminum (open circles) or titanium (closed circles). (Ref. 16.3)

Figure 49. The effect of temperature on TiAl and TiAl+Nb yield strength. (Ref. 178)

Figure 50. Effect of TiB₂ particles on colony size of TiAl ingots. Note rapid decrease in colony size with small addition of TiB₂. (Ref. 181)

Figure 51. Variation in TiAl creep rate with time at 854°C and 80 MPa. Dashed line is data for annealed sample. Solid line is unannealed material, with nitride formation during creep loading. (Ref. 191)

Figure 52. Particle size distribution of TiAl powders studied in this thesis.

Figure 53. Photograph of vacuum system used for degassing cans in preparation for HIP'ing, (A) roughing pump, (B) vacuum pump, (C) sample tubes.

Figure 54. Schematic representation of hot isostatic press (HIP).

Figure 55. Typical pressure and temperature profile during HIP cycle, with furnace cool.

3 of/de 3

PM 131 x4 PHOTOGRAPHIC MICROCOPY TARGET
NBS 1010a ANSI/ISO #2 EQUIVALENT

1.0

0.8

2.5

1.2

2.2

1.6

2.0

1.1

1.8

1.8

1.25

1.4

1.6

Figure 56. Tensile sample geometry for ambient and elevated temperature tests. Sample in lower photograph produced by grinding method outlined in Appendix B. (Hatch marks are at 1 cm intervals.)

Figure 57. General arrangement of test set-up for ambient and elevated temperature tensile tests.

GROOVE GEOMETRY

- all dimensions in mm.
- height to diameter ratio $\sim 1:5$
- end faces grooved to retain liquid lubricant

Figure 58. Geometry of specimen used for isothermal constant strain rate compression tests.

Figure 59. High temperature isothermal forging test set-up, showing;

- A induction heated furnace, with compression tooling inside,
- B hydraulic 100kN test frame,
- C analog function generator for constant strain rate tests,
- D proportional temperature controller,
- E induction high frequency generator,
- F vacuum and inert gas purging system.

- 1 load cell
- 2 plexiglass window
- 3 LVDT housing
- 4 quenching arm (TZM)
- 5 304 SS collar
- 6 cooling coil
- 7 induction coil
- 8 alumina transducer probes
- 9 SiN compression dies
- 10 TZM collar
- 11 TZM anvil
- 12 TZM or SS304 susceptor
- 13 quartz furnace tube
- 14 refractory plate
- 15 refractory rings
- 16 SS 304 collar
- 17 viton O-ring
- 18 347 SS anvil support
- 19 viton O-ring
- 20 water cooling passages
- 21 quenching chute
- 22 K type thermocouple
- 23 hydraulic actuator

Figure 60. Schematic cross section of high temperature isothermal compression testing facility.

Figure 61. Calibration chart for constant strain rate compression tests. Test time $t = \epsilon / \dot{\epsilon}$.

Figure 62. Instrumentation/control schematic for constant strain rate compression tests.

Figure 63. Cooling rate of compression platen from 1200°C, when flushed with argon.

Figure 64. Scanning electron micrograph of Ti-54%Al powder sample, as-received from Crucible Compaction Metals Inc. Note numerous satellites and misshapen particles.

Figure 65. Scanning electron micrograph of fractured Ti-54%Al powder particle. Note visible dendritic structure (A) and spherical particles (B).

Figure 66. Microstructure of titanium gas atomized Ti-54%Al powder.

Figure 67. Ti-54%Al as-received powder. Lamellar regions (A) are $\alpha_2 + \gamma$, light area (B) is interdentritic γ phase and area (C) is retained α_2 transformed from α dendrites.

Figure 68. Ti-54%Al as-received powder illustrating internal voids due to solidification shrinkage.

Figure 69. Scanning electron image of Ti-48%Al-2%W powder as-received from Crucible Compaction Metals Inc. Note larger proportion of small particles present compared to Ti-54%Al powder of Figure 64.

Figure 70. Backscattered electron image (BEI) of as-received Ti-48%Al-2%W powder. Note primary dendrites (A) and secondary dendrites (B).

Figure 71. Optical microstructure of as-received Ti-48%Al-2%W powder showing dendritic regions and peritectic α phase (arrows).

Figure 72. Backscattered electron image of Ti-48%Al-2%W as-received powder. Note composition difference between primary β dendrite (transformed to α'_2) and peritectic α surrounding dendrite.

Figure 73. EDS analysis for Ti-48%Al-2%W as-received powder. (A) spectrum for dark regions of Figure 72 corresponding to γ phase and, (B) spectrum for bright dendritic region corresponding to α/α_2 phase.

Figure 74. Unetched section of Ti-48%Al-2%W as-received powder. Note voids concentrated near surface of particle.

Figure 75. Microstructure resulting from HIP'ing Ti-54%Al at 1025°C/200 MPa/2 hr. Note presence of porosity (labelled P) and coring (labelled C) in (A) and prior particle boundaries and porosity in (B).

Figure 76. Microstructure of Ti-54%Al after HIP'ing at 1100°C/200 MPa/2 hr. Note wide range in grain size in (A) and recrystallization twins (arrows) in (B).

Figure 77. Microstructure of Ti-48%Al-2%W HIP'ed at 1050°C/200 MPa/2 hr. Note presence of well defined prior dendritic regions in (A). Prior particle boundaries (open arrows) and porosity (closed arrow) are evident in (B).

Figure 78. Grain structure of Ti-48%Al-2%W HIP'ed at 200 MPa and 2 hr. at temperatures; (A) 1050°C, (B) 1100°C, (C) 1150°C and (D) 1250°C.

Figure 79. Microstructure of Ti-48%Al-2%W HIP'ed at 1250°C/200 MPa/2 hr. (A) Optical micrograph, (B) SEM secondary electron image of etched region, (C) same area as in (B) but backscattered electron image, (D) Backscattered electron image of unetched region.

EDS Analysis of (A) dark equiaxed grains of Fig. 79C, corresponding to γ phase.

A

EDS Analysis of (B) small lighter equiaxed grains of Fig. 79C, corresponding to α_2 .

B

EDS Analysis of (C) bright phase at grain boundaries and triple points in Fig. 79C (next page).

Figure 81. EDS analysis of (A) dark equiaxed grains of Fig. 79C, corresponding to γ phase, (B) small lighter equiaxed grains of Fig. 79C, corresponding to α_2 and (C) bright phase at grain boundaries and triple points in Fig. 79C (next page).

C

Figure 81C. EDS analysis of bright phase at grain boundaries and triple points in Fig. 79C.

Figure 82. Transmission electron micrograph of interdendritic recrystallized region of Ti-48%Al-2%W HIP'ed at 1250°C/200 MPa/2 hr.

Figure 83. Microstructure of interdendritic region of Ti-48%Al-2%W HIP'ed at 1050°C. (A) Backscattered electron image of etched area. (B) Transmission electron micrograph showing deformation twinning and dislocations.

Figure 84. Backscattered electron image of 1050°C HIP'ed Ti-48%Al-2%W (unetched). Note compositional gradients in interdendritic region similar to Figure 70. Prior dendritic region indicated by 'P'.

Figure 85. (A) Transmission electron micrograph of interdendritic region of 1050°C HIP'ed Ti-48%Al-2%W, showing α_2 located at deformation twin boundaries. (B) Selected area diffraction pattern of the α_2 , γ and γ -twin phases shown in (A).

Figure 86. (A) β phase growing from α_2 lamellae in Ti-48%Al-2%W HIP'ed at 1050°C. (B) Micrograph of 3rd phase particles located along interfaces of 1050°C HIP'ed Ti-48%Al-2%W.

Figure 87. Backscattered electron image of 1050°C HIP'ed Ti-48%Al-2%W. Arrows indicate tungsten rich bands. (Unetched)

Figure 88. Migrating grain boundary (open arrows) demonstrating dynamic recrystallization during HIP'ing of Ti-48%Al-2%W at 1050°C. Note difference in dislocation density in recrystallized and non-recrystallized grains. Grain boundary migration is pinned by 3rd phase particle (closed arrows).

Figure 89. Recrystallization twin with 'ledge' type structure in 1100°C HIP'ed Ti-48%Al-2%W.

Figure 90. Dislocations in 1050°C HIP'ed Ti-48%Al-2%W, identified by diffraction contrast experiments as: $\rightarrow$ $\frac{1}{2}[\bar{1}\bar{1}2]$ superdislocation, $\blacktriangleright$ $[\bar{1}01]$ superdislocation, $\blacklozenge$ $\frac{1}{2}[1\bar{1}0]$ unit dislocation, and $\diamond$ $\frac{1}{2}[110]$ unit dislocation.

Figure 91. Plot of grain size and 'effective' grain size versus HIP'ed temperature for Ti-48%Al-2%W.

Figure 92. Microstructure of HIP'ed prior dendrites in TiAl+W. (A) Backscattered electron image of 1250°C HIP'ed material. Labels refer to EDS results of Figure 94. (B) Transmission electron micrograph of 1050°C material. Note absence of dislocations.

Figure 93. Relationship between dendrite area fraction and grain size with HIP temperature for TiAl+W.

Figure 94. EDS analysis for three areas identified in Figure 92a.

Figure 94. (continued) EDS analysis for areas identified in Figure 92a.

Figure 95. Optical micrographs of prior dendrites in TiAl+W HIP'ed at (A) 1050°C and (B) 1250°C.

Figure 96. Hardness versus HIP'ed temperature for Ti-54%Al and Ti-48%Al-2%W.

Figure 97. Comparison of the microhardness of prior dendritic and interdendritic regions of Ti-48%Al-2%W powder and HIP'ed material.

Figure 98. Room temperature engineering stress-strain curves for HIP consolidated Ti-48%Al-2%W.

Figure 99. Room temperature tensile fracture surface of Ti-48%Al-2%W HIP'ed at 1250°C.

Figure 100. Fracture surface of room temperature tensile tested Ti-48%Al-2%W, HIP'ed at 1150°C. Failure occurred in shoulder radius of gauge length, initiated by void indicated by arrow.

Figure 101. Example of stress-strain curves generated by duplicate tests of Ti-48%Al-2%W, HIP'ed at 1250°C and tensile tested at 850°C.

Figure 102. Longitudinal section of tensile sample, showing fracture path (indicated by arrows) of Ti-48%Al-2%W HIP'ed at 1050°C and tensile tested at room temperature.

Figure 103. (A) Tensile failure of Ti-48%Al-2%W HIP'ed at 1150°C and tested at 850°C. Note sperical surfaces of prior dendrites. (B) Longitudinal section of tensile sample shown in (A). Note failure and secondary cracking along prior particle boundaries.

Figure 104. Non-uniform deformation microstructure of 1100°C HIP'ed Ti-48%Al-2%W, tested at room temperature. (A) Dislocation free prior and interdendritic region. (B) Interdendritic region with high dislocation and deformation twin density.

Figure 105. Deformation structure of Ti-48%Al-2%W HIP'ed at 1250°C and tested at room temperature.

Figure 106. Deformation twin structure of 1250°C HIP'ed TiAl+W, tested at room temperature.

Figure 107. Development of deformation twins from an array of $1/6\langle 112 \rangle$ partial dislocations.

Figure 108. (A) A small dislocation free grain of α_2 (arrow) in TiAl+W HIP'ed at 1250°C and tested at 850°C. (B) Corresponding diffraction pattern of α_2 and γ .

Figure 109. Dislocation free grains (arrows) in TiAl+W HIP'ed at 1250°C and tested at 850°C.

Figure 110. $\frac{1}{2}\langle 110 \rangle$ (open arrows) and $\frac{1}{2}\langle 112 \rangle$ (closed arrows) dislocations in TiAl+W HIP'ed at 1250°C and tested at 850°C.

Figure 111. The effect of HIP temperature on the fracture strength of TiAl+W.

Figure 112. Isothermal compression sample, before compression and after ≈ 0.8 true strain. Hatch marks are 1 mm apart.

Figure 113. Quarter section optical micrograph of TiAl+W HIP'ed at 1050°C and isothermally compressed to ≈ 0.8 true strain at 900°C and 10^{-3} s^{-1} . Compression direction is horizontal, with sample/platen contact surface on right side of figure.

Figure 114. Photograph of TiAl+W HIP'ed at 1250°C and compressed at 1000°C and 10^{-1} s^{-1} , illustrating a radial crack. Hatch marks are 1 mm apart.

Figure 115. Schematic showing geometry of centre-line cracks observed in some compression samples (see Table XIV).

Figure 116. Micrograph of TiAl+W HIP'ed at 1050°C and compression tested at 1000°C and 10^{-1} s^{-1} to ≈ 0.05 true strain. Note cracking along prior powder particle boundaries and secondary radial cracks.

Figure 117. Load-height data generated during isothermal compression of TiAl+W HIP'ed at 1050°C and tested at 1100°C and 10^{-2} s^{-1} to ≈ 0.8 true strain.

Figure 118. True stress-true strain results for TiAl+W HIP'ed at 1050°C, showing the four generic curve types of stress strain curves obtained.

Figure 119. Comparison of repeatability of isothermal compression data for TiAl+W HIP'ed at 1250°C and tested at 1000°C.

Figure 120. Plots of isothermal compression behaviour of TiAl+W HIP'ed at 1050°C showing (A) the effect of temperature at a strain rate of 10^{-3} s^{-1} , and (B) the effect of strain rate at a temperature of 1100°C.

Figure 121. Plot showing the effect of HIP temperature on the isothermal compression behaviour of TiAl+W tested at 1100°C.

Figure 122. Optical micrograph of TiAl+W HIP'ed at 1050°C and tested at 1200°C and 10^{-4} s^{-1} , showing improved microstructural homogeneity due to isothermal compression. Compression direction is horizontal.

Figure 123. Dark field optical micrographs of TiAl+W HIP'ed at 1050°C and isothermally compressed. Note prior particle boundaries (arrows) discernable by change in β phase density. (A) Tested at 1100°C and 10^{-1} s^{-1} , with similar grain size in adjacent particles. (B) Tested at 1200°C and $3 \times 10^{-4} \text{ s}^{-1}$ with much larger grains in particle with lower β density. Compression direction is horizontal. Prior dendrites indicated by 'P'.

Figure 124. TiAl+W HIP'ed at 1050°C and isothermally compressed at 900°C/ 10^{-1} s $^{-1}$ to ≈ 0.8 true strain. Note profuse deformation twinning and small recrystallized grains (arrows).

Figure 125. TiAl+W HIP'ed at 1050°C and isothermally compressed at 900°C/ 10^{-1} s⁻¹ to ≈ 0.8 true strain. (A) Sample tilted to image all dislocations, and (B) sample tilted so that only superdislocations are imaged.

Figure 126. TiAl+W HIP'ed at 1050°C and isothermally compressed at 1100°C/10⁻¹ s⁻¹ to ≈0.8 true strain, exhibiting an almost completely recrystallized structure.

Figure 127. Dark field optical micrograph of TiAl+W HIP'ed at 1050°C and isothermally compressed at 1100°C/10⁻³ s⁻¹ to ≈0.8 true strain.

Figure 128. Structure of TiAl+W HIP'ed at 1050°C and isothermally compressed at 1200°C/ 10^{-3} s $^{-1}$ to ≈ 0.8 true strain, comprised of (A) recrystallized grains and (B) orderly dislocation arrays.

Figure 129. Finer grained prior dendritic region of TiAl+W after HIP'ing at 1050°C

Figure 130. Dark field optical micrograph of TiAl+W HIP'ed at 1050°C and (A) isothermally compressed to ≈ 0.8 true strain at $1200^{\circ}\text{C}/10^{-4} \text{ s}^{-1}$ and (B) heat treated at 1200°C for 8700 seconds. Both samples have same exposure time at 1200°C .

Figure 131. Relationship between peak true stress during isothermal compression and applied strain rate for TiAl+W HIP'ed at both 1050°C and 1250°C.

Figure 132. Contours of constant strain rate sensitivity exponent for TiAl+W HIP'ed at (A) 1050°C and (B) 1250°C as a function of applied strain rate and temperature. Circles indicate temperature/strain rate combinations tested (see Table XVI).

Figure 133. Dependence of strain rate sensitivity exponent on the applied true strain for TiAl+W HIP'ed at 1050°C and compressed at a strain rate of (A) 10^{-1} s^{-1} and (B) 10^{-4} s^{-1} . Curve type notation refers to stress-strain curve types shown in Figure 118. (Curve type 3 not observed for 1250°C HIP'ed material, see section 6.1.2.)

Figure 134. Dependence of strain rate sensitivity exponent on the applied true strain for TiAl+W HIP'ed at 1250°C and compressed at a strain rate of (A) 10^{-1} s^{-1} and (B) 10^{-4} s^{-1} . (Curve type 3 not observed for 1250°C HIP'ed material, see section 6.1.2.)

Figure 135. Linear relationship between peak true stress occurring during isothermal compression and inverse of temperature for TiAl+W HIP'ed at (A) 1050°C and (B) 1250°C.

Figure 136. Contours of constant activation energy Q_e (in kJ/mole) as a function of strain rate and test temperature for TiAl+W HIP'ed at (A) 1050°C and (B) 1250°C. Single digit numbers identify each test condition and the corresponding curve type according to Figure 118.

A **B**

Figure 137. Forgeability map identifying regions of *poor*, *suspect* and *good* forgeability as a function of strain rate and temperature for TiAl+W HIP'ed at (A) 1050°C and (B) 1250°C.

Figure 138. Strain rate sensitivity exponent as a function of applied true strain and corresponding grain sizes after ≈ 0.8 true strain for TiAl+W HIP'ed 1050°C. Initial HIP'ed grain size is $\approx 5 \mu\text{m}$ (see section 5.1.2.1). Microstructure after isothermal compression at 1100°C and 10^{-1} s^{-1} is shown in Figure 123a. Microstructure after isothermal compression at 1200°C and 10^{-4} s^{-1} is shown in Figure 130a.

Figure 139. Deformation map illustrating predominant deformation mechanism occurring at the peak true stress during isothermal forging as a function of applied strain rate and test temperature of TiAl+W HIP'ed at (A) 1050°C and (B) 1250°C. Dotted lines are contours of constant activation energy. SUBST - substructure development; DRX - dynamic recrystallization; DRX-SP - dynamic recrystallization and superplasticity; SP-GC - superplasticity and grain coarsening.

APPENDIX A

DERIVATION OF ACTIVATION ENERGY Q_e

This appendix gives the steps involved to obtain the stress, temperature and strain rate unique activation energy Q_e , discussed in section 2.3.3.1.

An equation to approximate the true stress-true strain behaviour is:

$$\sigma = \sigma(\dot{\epsilon}, T, \epsilon) \quad \dots\dots (A1) .$$

This does not strictly represent a constitutive equation, since plastic strain (ϵ) is a geometric rather than state variable. Rewriting this equation in differential form yields:

$$d \ln \sigma = \gamma d\epsilon + m d \ln \dot{\epsilon} + \phi dT \quad \dots\dots (A2)$$

The various coefficients are given by the partial derivatives:

$$\begin{aligned} \gamma &= \left[\frac{\partial \ln \sigma}{\partial \epsilon} \right]_{\dot{\epsilon}, T=\text{constant}} \\ m &= \left[\frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right]_{\epsilon, T=\text{constant}} \\ \phi &= \left[\frac{\partial \ln \sigma}{\partial T} \right]_{\dot{\epsilon}, \epsilon=\text{constant}} \end{aligned} \quad \dots\dots (A3) .$$

An assumption is made that the flow stress for a constant microstructure depends on Z , the Zener-Hollomon parameter (equation 6, section 2.3.3.1) which in turn depends on temperature and strain rate. Hence, ϕ can be written as:

$$\phi = \left[\frac{\partial \ln \sigma}{\partial \ln Z} \right]_{\epsilon, T} \cdot \left[\frac{\partial \ln Z}{\partial T} \right]_{\dot{\epsilon}, \epsilon} \quad \dots\dots (A4) .$$

The Zener-Hollomon parameter is defined as:

$$Z = \dot{\epsilon} \exp (Q/RT) \quad \dots\dots (A5) .$$

Taking the logarithm of equation A5 and differentiating with respect to temperature gives:

$$\frac{\partial \ln Z}{\partial T} = \frac{\partial \ln \dot{\epsilon}}{\partial T} - \frac{Q}{RT^2} \quad \dots\dots (A6) .$$

Note that the last term of equation A4 is evaluated at constant strain rate, thus equation A6 reduces to:

$$\frac{\partial \ln Z}{\partial T} = -\frac{Q}{RT^2} \quad \dots\dots (A7) .$$

Taking the natural logarithm of equation A5 and substituting it into equation A4 gives:

$$\frac{\partial \ln \sigma}{\partial \ln Z} = \frac{\partial \ln Z}{\partial (\ln \dot{\epsilon} + Q/RT)} \quad \dots\dots (A8) .$$

Once again, since equation A8 is evaluated at constant temperature, $\partial(Q/RT)=0$, so:

$$\left[\frac{\partial \ln \sigma}{\partial \ln Z} \right]_{\epsilon, T} = \left[\frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right]_{\epsilon, T} \quad \dots\dots (A9) ,$$

which is equivalent to the strain rate exponent, m , of equation A3. Hence:

$$\left[\frac{\partial \ln \sigma}{\partial \ln Z} \right]_{\epsilon, T} = m \quad \dots\dots (A10) .$$

Substituting equations A10 and A7 into A4, gives:

$$\phi = -m \frac{Q}{RT^2} \quad \dots\dots (A11) .$$

Substituting ϕ from equation A3 into equation A11 and rearranging, finally gives;

$$Q_{\epsilon} = \left[\frac{R}{m} \cdot \frac{d \ln \sigma}{d(1/T)} \right]_{\epsilon = \text{constant}} \quad \dots\dots (A12) .$$

which is equation 7 of section 2.3.3.1.

APPENDIX B

TENSILE SAMPLE FINISH MACHINING PROCEDURE

Mechanical test samples were finish machined utilizing a procedure of low stress grinding followed by polishing. This appendix outlines this operation, as applied by Stulen Machine Inc., Pittsburgh, Penn.

LOW STRESS GRINDING

Specimens are contour ground in three steps, a rough grind of 0.003" and two finish grinding operations of 0.002". Grinder speed is 3400 rpm and head stock speed is 650 rpm. The horizontal feed is 1" in three minutes and a 4" diameter 'HA-120-06-Vio' grinding wheel is used. Lubrication is 'Veedol Afton 8'. Specimens are ground to 0.0015" oversize to allow for final polishing.

POLISHING

A semi-automatic polishing machine is used in which the specimen can be rotated. Abrasive tape of ½" wide 180 grit aluminum oxide is fed around a 3" diameter rubber roll directly above the test specimen. The rubber roll is brought into contact with the test specimen with a controlled and constant pressure. The abrasive tape is fed across the specimen at 8" per minute and then discarded. An arm carrying the rubber roll and abrasive tape reciprocates in a direction parallel to the specimen length. The grain of the abrasive tape is in contact with the specimen for 1 minute, ensuring free cutting action of each abrasive grain. The specimen is rotated at a speed so as to produce a crosshatch scratch pattern. This process is repeated using 400 grit abrasive, maintaining the crosshatch pattern.

The sample is the finished to size using 600 grit abrasive bonded to a 0.625" roll and used only once. A longitudinal scratch pattern is maintained. A good grade of lapping oil is used for lubrication (LMKT Sperm oil substitute is recommended). This final operation produces a polished surface with no circumferential scratches visible at 40 times magnification.

APPENDIX C

TiAl+W ISOTHERMAL CONSTANT STRAIN RATE

TRUE STRESS-TRUE STRAIN RESULTS

END

1 2 - 1 2 - 9 3

FIN