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**THE CREEP BEHAVIOUR OF LAMELLAR
NEAR γ -TITANIUM ALUMINIDES**

by

JIM TRIANTAFILLOU, B.ENG.

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

Master of Engineering

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

**Carleton University
Ottawa, Ontario
October 1996**

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ISBN 0-612-19510-4

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ABSTRACT

The effect of microstructure on the creep resistance of Ti-48%Al is presented. The microstructural parameters analyzed include: the volume fraction of γ grains, the lamellar interface spacing and the grain boundary morphology. Increasing the volume fraction of γ grains increases the creep strain rate and fracture strain, while creep life decreases. Creep deformed structures indicate that grain boundary sliding and dynamic recrystallization may be factors contributing to the early onset of tertiary creep for structures with high γ grain volume fractions.

Results from tests to determine the effect of lamellar interface spacing on creep properties, indicate that the minimum creep strain rate is increased by a factor of 5 when the average interface spacing is increased. Results from stress dependent tests suggest that dislocation creep processes control the creep deformation at 240 MPa and 760°C. Interlocked lamellae along grain boundaries improve resistance to intergranular cracking, therefore allowing greater total creep strain and potentially greater creep life.

ACKNOWLEDGMENTS

I would like to extend my appreciation to Dr. J. Beddoes, for his help throughout this thesis. His determination and enthusiasm were encouraging to myself and others involved in similar research. I owe Dr. J. Beddoes a considerable gratitude for assistance in interpretation of results but as well an even greater debt is owed to him for help in obtaining my current employment position.

I wish to thank Carleton University and the National Research Council Canada for financial assistance. The help of D. Morphy and L. Zhao is particularly appreciated. I would also like to thank W. Chen for help with transmission electron microscopy.

Above all, I wish to thank my family for unending support and patience during the last few months. In particular I would like to thank Anna who has provided me with constant support and caring made all of this possible.

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NOMENCLATURE

symbol	description
A	constant dependent on microstructural from power law creep equation
AC	air cooled
b	Burger's vector
d	grain diameter for Nabarro Herring and Coble creep relations
DP	duplex
DO ₁₉	representation of alpha-2 crystal structure
DRX	dynamic recrystallization
D _{SD}	diffusion coefficient for lattice self diffusion
FC	furnace cooled
FCC	face centered cubic
FCT	face centered tetragonal
FL	fully lamellar
G	shear modulus
g.b.'s	grain boundaries
GBS	grain boundary sliding
HCP	hexagonal close packed
HIP'ed	hot isostatically pressed
L	gamma lamellar spacing

Ll_0	representation of gamma crystal structure
LGS	lamellar grain size
LS	average lamellar interface spacing
NL	nearly lamellar
Q	activation energy
R	universal gas constant
T	temperature
TiAl	titanium aluminide

α	alpha phase with hexagonal close packed crystal structure
α_2	alpha-2 phase with hexagonal close packed crystal structure
γ	gamma phase with face centered tetragonal crystal structure
$\dot{\epsilon}_{min}$	minimum creep rate
ν	mobility of climbing dislocations
τ_o	Orowan shear stress
Ω	volume of a vacancy

CHAPTER I

I. Introduction

During the last twenty years, an increasing research and development effort has been directed toward near γ -titanium aluminide intermetallics. These intermetallic compounds contain between 45 and 50 atomic percent aluminum and potentially offer a combination of properties useful for several gas turbine and automotive engine applications. Along with a low density, near- γ titanium aluminides (γ -TiAl) also have a high modulus of elasticity, high melting point and good creep resistance essential for high temperature use[1].

To understand specific titanium aluminide alloys it is first necessary to understand what constitutes an intermetallic compound. Intermetallic compounds are formed when a specific solute element added to a host metal at certain compositions results in solid solution properties that differ considerably from the constituent elements. These differences are that the intermetallic compound has a highly ordered atomic structure with a crystal structure that is different from its constituent elements. Intermetallic structures possess lower energy unlike bonds than like bonds leading to A-B rather than A-A or B-B bonding[2]. The ordered structure results in low diffusivities within the lattice structure and consequent superior high temperature performance.

Table 1 [1] compares the properties of near γ -TiAl intermetallics with Ti-alloys and Ni base superalloys. From a comparison of these intermetallics to current alloys several areas of improvement are noted, particularly with regard to density and high temperature resistance with increased performance noted for several properties In

particular, current superalloys have densities of approximately 8.3 g/cm^3 while the density of alloys based on titanium aluminides range from 3.7 to 4.7 g/cm^3 .

1.1 Applications for Titanium Aluminides

Several gas turbine applications can benefit from the low density and high temperature capability of near γ -TiAl. The high temperature strength coupled with low density will lead to increased efficiency and an improved thrust to weight ratio. Conventional titanium alloys are restricted from use in hot engine parts because of poor burn resistance and oxidation at high temperatures ($>590^\circ\text{C}$). In contrast, near γ -TiAl can be used at up to 815°C without extensive oxidation and with good burn resistance.

As well as low density and high temperature strength, near γ -TiAl also provides a considerable increase in specific stiffness (E/ρ) compared to current alloys (Inconel 718), if used in engines for the same proposed applications. The increase in stiffness is approximately 50 percent, which is valuable in areas of tight tolerance such as framing members and compressor casings. Other parts fabricated of near γ -TiAl for use in aero engines could be low pressure turbine blades, turbine frame and fan sections. Low pressure (LP) turbine blades represent the application with the highest payoff for using a near γ -TiAl alloy, but it is also the highest risk. General Electric has tested a full set of 5th stage LP blades in the 60000 lb. thrust CF6-80C engine for over 1000 full throttle cycles without any significant signs of wear or degradation on the blades, Figure 1 shows these blades in the pre and post test conditions[3]. A recent application suggested for near γ -TiAl are low pressure turbine blades for the GE90 turbo fan designed for the 777 aircraft, which is the next generation of high thrust engine for use in commercial

transports. The potential weight savings from using near γ -TiAl for this one application is approximately 300 lb.[4].

In addition to aerospace applications, near γ -TiAl has been proposed for use in the automotive industry, for exhaust valves and turbo charger rotors[5]. For applications in the valve train the lower density leads to a quieter, more fuel efficient engine with higher performance. These improvements arise due to the lower weight of the valves allowing a lower spring tension to close them, thereby reducing friction in the valve train. As well the higher temperature capability of these materials compared to steel valves is noted[6]. Also, performance increases, with the high speed capability of the valve train increased by 600 rpm by using near γ -TiAl valves, leading to a potential power increase of approximately 5 percent[7]. Both GM and Ford have tested near γ -TiAl exhaust valves for several years. A GM vehicle has gone 50000 miles without any reported problems on a full set of near γ -TiAl valves, with no reported failures during testing, from either GM or Ford[6]. The main obstacle to overcome for exhaust valve applications is not the performance of the valves, but rather the development of an economically feasible fabrication process to produce quality valves that meet specifications[5]. Some manufacturing technologies currently under investigation are, investment casting and permanent mold casting, with examples of valves produced from these processes shown in Figure 2 a and b and a machined finished valve in figure2 c[7].

1.2 Thesis Objectives

As mentioned, near γ -TiAl intermetallics will most likely find use in the aerospace or automotive industries where their lightweight and high temperature

capabilities are best utilized. Of particular importance during high temperature service of aerospace gas turbine components are the elevated temperature strength, fracture toughness, and creep resistance. Creep resistance becomes crucial in the hot zones of gas turbines where tolerances are tight between blades and casings. Excessive creeping of blades under the action of high radial loads could cause failure of an engine. The improvement of creep resistance has been identified as a major barrier to increasing the service temperature of near γ -TiAl alloys[6]. The creep properties are controlled by alloy composition, processing conditions and microstructure. It has been demonstrated that creep properties of near γ -TiAl are improved by the development of a lamellar microstructure consisting of large grains composed of alternating lamellae of the α_2 and γ phase[8]. However, there has been minimal work examining the influence of the individual lamellar microstructural parameters present in these lamellar type of microstructures and the resultant creep behaviour. It is, therefore, the objective of this thesis to evaluate the creep behaviour of several predominantly lamellar microstructures with varying microstructural parameters, namely the volume fraction of γ grains, lamellar interface spacing, and grain boundary morphology. This work will contribute to an improved capability for microstructural design for specific applications.

For the investigation of the creep behavior of these microstructures, several tasks have been completed:

- 1) Review of literature for near γ -TiAl with emphasis on current research and development relevant to the current investigation.
- 2) From the knowledge obtained in review of current literature, heat treatments were developed to obtain a range of γ grain volume fractions, interface spacings and grain boundary morphologies.

- 3) Microstructural characterization of the resultant microstructures was carried out using optical and electron microscopy.
- 4) Creep testing of microstructures was done with constant load creep equipment. Creep tests were undertaken with monotonic loads, incremental loads, as well as using the consecutive stress reduction technique. Optical and electron microscopy were carried out on the deformed specimens to determine the deformation mechanisms associated with the creep response.

A review of the current literature relevant to this thesis work is provided in Chapter 2. Chapter 3 follows with a more thorough definition of the thesis objectives. Chapter 4 provides an outline of the Materials Preparation and Experimental Procedure. The results from task 3 and 4 are presented and discussed in Chapters 5 through 7. Summary and conclusions are outlined in Chapter 8.

Although considerable previous work deals with creep of near γ -TiAl intermetallics, much of it has focused on ternary or quaternary alloys of near γ -TiAl compositions with the aim of producing a material for rapid introduction to service. The aim of this work is to obtain a better understanding of the role microstructure has on creep properties, while still maintaining the objective of determining microstructural parameters for optimal mechanical property response. Consequently, for this thesis, a binary titanium 48 atomic percent aluminum alloy is investigated. To accomplish this a thorough understanding of past and current work is presented, dealing with fundamental crystallography, microstructural evolution through heat treatment, microstructure/mechanical property relationships, with an emphasis on creep properties related to microstructure, and finally creep fundamentals.

CHAPTER 2

2. BACKGROUND LITERATURE REVIEW

The following review will be broken down into sections covering fundamentals of near γ -TiAl which will include, crystallography, heat treatment options, mechanical property/microstructural relationships, and a final section on creep fundamentals.

2.1 Crystallography and Deformation of TiAl and Ti₃Al

Near γ -TiAl alloys consist of two phases, γ -TiAl and α_2 -Ti₃Al. The morphology of these two phases depends on the thermomechanical processing applied. However, both γ -TiAl and α_2 -Ti₃Al are intermetallic compounds with highly ordered crystal structures from which many of their physical and mechanical properties derive. A brief review of the crystallography of these intermetallics is presented, followed by a review of deformation related to the crystal structure.

γ -TiAl has the ordered face centered tetragonal (FCT) structure, I_1I_0 , illustrated in Figure 3 with the stoichiometric compound having a c/a ratio of 1.015, or a nearly cubic structure. As aluminum concentration increases the ratio of tetragonality increases to 1.03 and decreases to 1.01 with a decrease in aluminum concentration[9]. The FCT structure leads to the $\langle 110 \rangle$ and $\langle 011 \rangle^*$ directions not being equivalent as in an FCC structure, which leads to many mechanical property attributes[10]. In particular, due to

* The Miller indices notation $\langle hkl \rangle$ indicates that h and k may have any valid indice, but l cannot change.

this tetragonality, the FCT lacks the propensity for slip that other FCC structures possess. As well, γ -TiAl remains ordered up to the melting temperature of 1450°C.

The α_2 -Ti₃Al has the ordered hexagonal close packed, DO₁₉ atomic arrangement depicted in Figure 4. α_2 has a wide composition range from 22at% to 39at%Al[11]. The compound is congruently disordered at a temperature of 1180°C and an aluminum concentration of 32at%[11].

During processing of near γ -TiAl alloys the γ -TiAl phase develops from either the α -Ti or α_2 -Ti₃Al phase to form a lamellar structure α_2 and γ lamellae. The two phases have the relationship $(111)_\gamma // (0001)_{\alpha_2}$. That is, the γ phase forms on the basal plane of the HCP α_2 . This typically occurs on stacking faults in the HCP structure, since a change in stacking sequence of α or α_2 leads to a FCC arrangement which approximates the FCT structure of the γ phase. A more detailed explanation of the formation of γ in α or α_2 will follow in a later section.

The deformation behaviour of both γ and α_2 is related to their crystal structures. The slip plane for deformation of the γ phase are the $\{111\}$ planes with the lowest energy Burgers vectors on this plane, in order of increasing energy, being the $1/2\langle 110 \rangle$ unit dislocations, $1/2\langle 112 \rangle$, and $1/2\langle 101 \rangle$ superdislocations, Figure 3[9]. The superdislocations can undergo complex dissociation reactions, for example, $[101] \rightarrow 1/2[110] + 1/2[112]$ where the $1/2[112]$ may dissociate further according to $1/2[112] \rightarrow 1/6[112] + (\text{stacking fault}) + 1/3[112]$. After room temperature deformation, a large number of sessile faulted dipoles are observed. These are formed by dissociations of superdislocations resulting from pinning of $1/6\langle 112 \rangle$ partials[9]. Pinning is believed to be the result of a complicated cross-slip/redissociation mechanism[12]. These pinned

dislocations appear to control the room temperature strength and cause the resulting low ductility of γ -TiAl. In addition, the limited number of glide systems may contribute to the low room temperature ductility[13].

α_2 -Ti₃Al with the D0₁₉ structure has three linearly independent slip systems for motion of $1/6\langle 1210 \rangle$ type dislocations on the basal (0001), prism (1010) and pyramidal (0221) planes in Figure 4. There are two additional slip systems involving $\langle c+a \rangle$ slip with a Burgers vector of $1/6\langle 1216 \rangle$ on slip planes of (1211) and (0221). Basal type slip is the major deformation mode with most slip occurring being $1/6\langle 1210 \rangle$. The planarity of slip and the limited number of slip systems leads to the limited ductility and resulting cleavage fracture of the α_2 phase[11].

2.2 Heat Treatment Options and Related Microstructures

Alloys for structural use contain a mixture of γ or α_2 phases in a variety of microstructures. Depending on heat treatment conditions a range of microstructures is produced, each with differing mechanical properties. This section first deals with heat treatments and related microstructural development from consideration of the Ti-Al phase diagram, followed by a discussion on microstructural/property relations.

2.2.1 Phase Diagram for TiAl Alloy System

The Ti-Al binary phase diagram has only recently been revised to its current form (Figure 5a). For the work of this thesis the central portion of the phase diagram is of critical importance and is illustrated in an expanded view (Figure 5b). This central

portion is the key region of interest when dealing with alloys such as Ti-48%Al. As can be seen from Figure 5b, this composition is near the border of the γ region giving rise to designation as near γ -TiAl alloys.

In this central portion there are three solid phases consisting of γ -TiAl, α_2 -Ti₃Al and α -Ti, as well as two reactions: a peritectic $L + \alpha \rightarrow \gamma$ and a eutectoid $\alpha \rightarrow \alpha_2 + \gamma$. The line representing the transition from $\alpha \rightarrow \alpha_2 + \gamma$ is known as the α transus line. The peritectic reaction occurs at $\sim 1485^\circ\text{C}$ and 46% Al while the eutectoid reaction is at $\sim 1125^\circ\text{C}$ and 39% Al, as indicated in the phase diagram. Precise location of these boundaries is still under investigation. The eutectoid reaction rarely occurs due to the different transformation kinetics of the nucleation of γ and α_2 phases. The α -Ti phase is HCP which implies an easy transformation from α to α_2 which proceeds at small undercooling. The transformation $\gamma \leftrightarrow \alpha$ occurs with slower kinetics due to their different structures (HCP $\leftrightarrow$ FCT).

With portions of the binary phase diagram still under dispute most ternary phase diagrams are incomplete due to the same inability to place boundaries precisely. With the addition of ternary alloying elements there are significant changes to the phase diagram for TiAl. For example with the addition of chromium the α transus is shifted downwards as indicated in Figure 6[14]. Research has shown that the α transus decreases by 15°C with each atomic percent addition of chromium[14]. To date the most complete ternary system studied is the Ti-Al-Nb system, with this system still requiring more research to more accurately locate phase boundaries[15].

2.2.2 Microstructures

From the Ti-Al phase diagram for two phase alloys of the near γ -TiAl compositions several microstructures are developed depending on the thermal processing. For near γ -TiAl compositions four types of microstructures are generally obtained: near γ equiaxed, duplex, nearly lamellar and fully lamellar, which are shown in Figure 7. The following section reviews these microstructures with emphasis placed on the fully lamellar type.

2.2.2.1 Fully Lamellar Microstructures

Heat treatments above the α transus, temperature T_t in Figure 5b, result in the formation of the fully lamellar microstructure illustrated in Figure 7d. The fully lamellar microstructure consists of large grains ($>300 \mu\text{m}$) made up of alternating γ and α_2 plates that form during cooling from heat treatment in the α phase field. The growth of lamellar grains has been found to obey the grain growth law:

$$D=Kt^n \quad \text{.....(1)}$$

where D is the grain diameter, K is a constant, t is time, and n the growth exponent. From previous research the constants determined from a log-log plot of heat treatment time versus grain size for air cooled samples heat treated at 1400°C were $K = 70$ and $n=0.3$ while for furnace cooled samples the results were $K = 76$ and $n= 0.3$. These results suggest that the grain growth from above the α transus is independent of the cooling rate [16].

Although the lamellar grain size may not be cooling rate independent, the morphology of the lamellar structure and the formation of the lamellae is definitely cooling rate dependent. There are two recognized reactions that describe lamellar

formation in TiAl when heat treated above T_{α} , that lead to a fully lamellar microstructure.

The two reactions are [8]:

where L refers to the lamellar morphology.

In the first mechanism the transformation begins below the α -transus at approximately 1377°C where the α phase, begins to transform to the γ -TiAl, through an altering of the stacking sequence. The normal sequence in HCP is ABAB.... whereas in FCC (face centred cubic), used to approximate FCT since the c/a ratio is nearly 1.0 for γ -TiAl, the stacking sequence is ABCABC... where the (111) plane of the γ phase is oriented parallel to the basal (0001) plane of the α structure. Because there is only this one plane on which the transformation can occur, there is only one lamellar orientation per grain. The remaining α is then chemically ordered to α_2 at a temperature that has yet to be determined[17]. Evidence of this type of reaction has been observed from transmission electron microscopy studies. If a sample is cooled from the α phase field to the $\alpha+\gamma$ field (~1230°C) and held for 72 hours the resulting microstructure is fully lamellar. The lamellar structure is composed of well developed γ and α_2 lamellae with the α_2 lamellae containing anti phase boundaries (APB's) indicating γ formation occurs prior to $\alpha \rightarrow \alpha_2$ ordering[18]. This reaction occurs during cooling from the α phase field at a relatively slow rate ($\approx 30^\circ\text{C}/\text{minute}$).

The second mechanism of formation begins with ordering of the α to form α_2 . It has been proposed that the γ phase nucleates by the movement of Shockley-type partials which lead to an FCC type stacking sequence by a stacking fault mechanism in a manner similar to that described for the first mechanism[8]. This has been confirmed[18] by

transmission electron microscopy studies, which indicate that there is evidence of γ lamellae cutting through APB's, which verify that the α_2 phase was present before the formation of the γ lamellae. This type of reaction occurs completely when during cooling from the α phase field at a relatively fast rate.

2.2.2.2 Duplex and Nearly Lamellar Microstructures

Heat treating in the $\alpha+\gamma$ phase at temperatures where α and γ phases are approximately equal by volume, around T_2 in Figure 5b, results in a structure consisting of fine equiaxed γ grains, and lamellar type grains, Figure 7a. The lamellar grains result from transformation of some γ grains to α which on cooling form the previously discussed lamellar structure. The γ phase volume fraction is reduced by dissolution until the equilibrium volume fraction of each phase is reached. During the course of achieving equilibrium there is competitive grain growth between the two phases resulting in a fine microstructure of γ grains and lamellar type grains.[15] The volume ratio of γ to lamellar grains is dependent on the heat treatment temperature, but the transformation sequence remains unchanged and can be described, for a typical as HIP'ed (Hot isostatically pressed) microstructural condition, as:

When heat treating in the upper portion of the $\alpha+\gamma$ phase field near the α transus, (at T_3 in Figure 5b) the amount of α phase increases consistent with the phase diagram, and as a result larger lamellar grains are formed with less untransformed γ remaining, as seen in Figure 7c. The γ grains appear predominantly at lamellar grain boundaries, but by increasing the heat treatment time from 2 to 100 hours the γ grains become located within

larger lamellar grains (Figures 8a and b). This indicates that α grain boundary migration occurs regardless of the presence of γ grains[19].

2.2.2.3 Near γ Microstructure

A microstructure composed almost entirely of γ grains may be obtained, Figure 7a, by heat treating near the eutectoid temperature, for long periods of time (14 days), T_4 of the phase diagram in Figure 5b. Previous studies have shown that by increasing the heat treatment time further, the grain size is only marginally increased up to 110 μm for a heat treatment of 14 days at 1130°C. The reason for this restricted grain growth is attributed to the α_2 phase pinning the γ grain growth at the boundaries.

2.3 Morphology of Lamellar Structures

As mentioned previously, the formation of the lamellar structure is dependent on the cooling rate, with the variation of several lamellar microstructural parameters dependent on the exact heat treatment conditions. To understand the role these parameters may have on mechanical properties, the particular microstructural features are first described.

Studies have shown that the spacing between γ and α_2 plates, the lamellar interface spacing (LS), is a function of the cooling rate, Figure 9[17]. From Figure 9 it is obvious that the lamellar interface spacing decreases with increased cooling rate. This may suggest diffusion controlled formation of the lamellar structure. The nucleation of γ in α is higher at faster cooling rates, however as the cooling rate drops, diffusion slows down and the result is many finely spaced plates, Figure 10a. For a slower cooling rate,

there are fewer γ plates nucleated and the result is thicker plates due to the increased amount of time at the higher temperature facilitating more diffusion during cooling. Figure 10b[20]. If an extremely slow cooling rate is applied to a sample the $\alpha \rightarrow \gamma$ transformation proceeds in a near equilibrium manner, with γ grains beginning to form from the plates in order to reduce the interfacial energy between the two phases[17].

2.3.1 Secondary Lamellar Structures

If a rapid cooling rate (air cooling) is applied a Widmannstätten type of lamellar structure is observed, which will be referred to as secondary lamellar structures. These types of structures can be seen in Figure 10a. This formation often has an appearance similar to the primary lamellar structure within which it forms. It consists of alternating plates similar to those of the parent lamellar grain. If even more rapid cooling (water quench) is used, a massively transformed type of feature is often observed with a feathery mottled appearance, also evident in Figure 10a. The massive transformation by definition implies that a change occurs in crystal structure without a change in chemical composition. This type of reaction often occurs when pertinent phase fields overlap in composition, often below a metastable two phase field, such as a eutectoid temperature. This reaction takes place when other competing equilibrium transformation modes such as Widmanstätten formation.

The volume fraction of secondary structures has been found to increase with increasing Al content in the range of 46.5 to 50at%Al[21]. The formation of these structures is also dependent on the temperature from which they are cooled, with

specimens cooled from the α region being more likely to have secondary structures than those cooled from the $\alpha+\gamma$ region[21].

Several mechanisms of formation have been proposed[21] for the formation kinetics of secondary structures.

1) As samples are cooled at intermediate rates the temperature is continuously dropping and the γ plates nucleate out of the α grains at grain boundaries and grow into grains. As the sample becomes sufficiently undercooled the untransformed α is trapped in the grain interior. Once sufficiently undercooled it transforms massively to form the feathery mottled appearing microstructure. Although offering an explanation to the formation of the massive γ regions, this does not explain the formation of the Widmännstatten type of secondary lamellar regions.

2) As the cooling rate increases the lamellar morphology breaks down as it can't grow with speeds paralleling the heat transfer rates out of the system. Under these conditions γ may form and grow on semi-coherent/incoherent interface.

3) As the temperature drops thermal stresses develop that cause twin formation to occur in the α phase on (1121) and (1122) planes. Subsequent cooling results in differently oriented Widmännstatten type structures developing on the twins within primary lamellar grains.

None of these explanations fully accounts for the formation of secondary structures clearly and further study is needed to identify the formation mechanism .

2.3.2 Discontinuous Coarsening of Lamellar Structures

In addition to the secondary structures formed within the primary lamellar structure, another phenomenon observed is discontinuous coarsening in lamellar type microstructures. When comparing the microstructures from Figures 10a and b, the air and furnace cooled specimens with differing lamellar spacing, another important feature is the more serrated appearance of the furnace cooled grain boundary[20,22]. It has been suggested that the serrated grain boundaries are an early stage of discontinuous coarsening mechanism[22,23] which leads to a thickening of the lamellae at grain boundaries accompanied by grain boundary migration.

During discontinuous coarsening there is growth of the primary lamellar structure into adjacent grains forming a secondary lamellar structure with the same orientation as the parent lamellar grain. As this secondary lamellar structure continues to grow it often changes orientation forming secondary degenerate lamellae[24]. The growth of the lamellar structure within lamellar grains is by a "ledge" mechanism and due to the low density of ledges observed at the interphase interface the thickening rate is correspondingly slow. Therefore because of the highly coherent α_2/γ interfaces at interior grain sites, grain boundaries offer greater solubility which may promote diffusivity within the γ phase leading to the discontinuous coarsening as the mode for lamellar thickening[24]. Heat treatment time also has an effect on the discontinuous coarsening. Previous research has shown that for long-time heat treatments (100 hours), grain boundaries appear more planar than comparable heat treatments at shorter times (25 hours), Figure 11a and b[19]. It was suggested that after 100 hours at temperature (1375°C), α grain boundary energy is insufficient for γ lamellar growth into the

incoherent grain, while for the 25 hour condition there is sufficient energy for γ growth into the incoherent grain thereby lowering the total free energy of the system[19].

2.4 Microstructure -Mechanical Property Relationships

Depending on the microstructural condition of near γ -TiAl alloys, the mechanical response of the material is different. In this section, the effect of microstructure on mechanical properties will be reviewed for the previous microstructural conditions discussed. Tensile strength and ductility, fracture toughness, fatigue will be addressed leading to a more comprehensive look at creep of γ -TiAl alloys focusing on lamellar microstructures.

2.4.1 Tensile Properties and Ductility

The typical room temperature elongation for two phase near γ -TiAl alloys is between 0.3%-4% with yield and tensile strengths varying between 350-600 MPa and 300-700 MPa respectively depending on alloy composition, processing and microstructure[15,25]. The microstructure with the highest room temperature tensile ductility is the duplex microstructure, Figure 12, consisting of fine equiaxed γ grains and lamellar grains[15]. The coarser grained fully lamellar microstructure exhibits the lowest levels of strength and ductility in two phase near γ -TiAl, curve A, Figure 12. As the grain size decreases the strength and ductility increase as curves B and C represent fully lamellar specimens with decreasing grain size. An increase in strength and ductility is noted for the nearly lamellar condition, curve D, when compared to the fully lamellar

results, curves A, B, and C, Figure 12.[15] This variation can be attributed to the small γ grains present in both the duplex and nearly lamellar microstructures which generally leads to a higher yield strength and greater elongation.

In near γ -TiAl, tensile ductility is also dependent on temperature with a ductile-to-brittle transition temperature (DBTT), ranging from 600°C to 820°C depending on composition and microstructure[15,26]. The effect of temperature on tensile properties is shown for the various microstructural conditions in Figure 13[15]. From Figure 13, the duplex alloys exhibit lower DBTT while the fully lamellar microstructures have higher transition temperatures. The fully lamellar structures also show retention of strength at higher temperatures than the duplex microstructures[25]. Leading up to the DBTT ductilities increase marginally until the DBTT when ductility increases rapidly. This is attributed to greater twinning and ordinary dislocation activity[15,25,26].

2.4.2 Fracture Toughness and Fatigue

Fracture toughness relates a material's ability to resist the extension of a crack. For γ -TiAl alloys there is an inverse relationship between microstructure and fracture toughness to that of ductility. The fully lamellar microstructure features the best fracture toughness, with values for K_{IC} typically between 20-35 MPa $\sqrt{m}$, while the duplex alloys are within the range 10-16 MPa $\sqrt{m}$ [25]. Studies have shown that the duplex structure has small plastic strains at the crack tip and no resistance to cracking while the fully lamellar microstructure yields large crack-tip strains and offers more resistance to crack propagation. The fracture strain in the plastic zone for the fully lamellar condition can be 20% while only 3% for the duplex condition, Figure 14[25]. The difference between the

fracture toughness of the lamellar and duplex alloys can be explained by considering that there is redundant work expended in fracturing ligaments that are formed in the crack wake of lamellar microstructures, with a ligament shown in Figure 15[27]. Shear ligament toughening provides significant crack deflection and branching during fracture[27,28]. In duplex alloys there are no bridging ligaments present and therefore no resistance offered by the tortuous crack path seen in lamellar structures, Figure 15. It has also been suggested that the fully lamellar structure is tougher due to its anisotropic composite-like characteristics which serve to arrest cracks[26].

Low cycle fatigue life of duplex microstructures is typically longer than that of fully lamellar microstructures below 850°C[25], while at high cycle conditions, fully lamellar microstructures have better fatigue properties at higher temperatures, Figure 16a[25]. Fatigue crack growth rates for duplex microstructures are compared to those of fully lamellar in Figure 16b[25]. The crack growth rates are considerably improved for the fully lamellar microstructure at room temperature and at 800°C[30], which appears to be consistent with the previous review of fracture toughness. For the duplex structure crack initiation usually occurs at the specimen surface while for the fully lamellar condition, cracks initiate in the interior[29]. The causes of crack initiation are still under investigation.

2.4.3 Creep Properties

In this section the creep properties of γ -TiAl alloys will be reviewed based on microstructural modification of both single and multiphase alloys. The review of

multiphase alloys will present a more comprehensive review of microstructure and the effect of stress, since these types of structures are the subject of this thesis.

2.4.3.1 Creep of Single Phase Gamma TiAl

Single phase γ microstructures contain no α_2 phase and a more regular equiaxed structure is obtained since these compositions are in the γ phase field and there is no competing growth between phases. Although not being considered for applications they offer a considerable insight to the understanding of fundamental creep behaviour, which may be used for understanding two phase alloy behavior.

Studies dealing with creep of single phase γ microstructures based on modification of grain size and alloy composition have produced results shown in Figures 17-19[31-33]. The three alloys show a varied response depending on the specimen grain size ranging from 25-70 μm . For the Ti-53.4%Al there is no pronounced grain size effect at either stress level, Figure 17. The intermediate composition of Ti-51.5%Al, shows no grain size effect at 251 MPa, but at the lower stress of 100 MPa an effect is noted, Figure 18, for the grain size ranging from 22-46 μm . For Ti-50%Al alloy a grain size effect (with grain sizes ranging from 32-170 μm) was observed even at 251 MPa, Figure 19. These results indicate that the effect of grain size is a factor only when aluminum content is lowered and stress is decreased. By plotting the minimum creep rates obtained from Figures 17-19, versus the stress levels tested, three regions are identifiable as seen in Figure 20. In region I, dynamic recrystallization controls the minimum creep rate. Small recrystallized grains were visible at grain boundaries with the total volume of these new grains increasing with the strain[34]. In region II of Figure 20 the minimum creep rate is the result of a balance between the slip of ordinary dislocations and superdislocation

activity leading to hardening, and softening due to grain boundary recrystallization[34]. In region III there is similar dislocation activity as in region II, but because of the low strains there is sufficient time for climb of these dislocations to cause sufficient recovery so that no dynamic recrystallization occurs[34]. A general observation can be made that as the aluminum content decreases the minimum strain rate decreases.

The power law creep equation is often used to help explain the fundamental processes occurring during creep. The equation is,

$$\dot{\epsilon}_{\min} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad \dots(1)$$

where A is a constant dependent on the microstructure, σ the stress, n is the stress exponent, Q the activation energy, R the universal gas constant, and T is the absolute temperature. The previous results have been described as being within the power-law creep regime[35]. The stress exponents for regions I, II and III are 4.7, 7.5 and 3.5. The creep mechanism predicted by the power law stress exponents 'n' imply diffusional controlled creep for exponents of ≈ 1 , while dislocation climb controlled creep is indicated for exponents of $\approx 3-5$ [36]. From the results of the previous investigation[31-35] the exponents determined from the stress dependence of the minimum creep rate, Figure 20, are higher and do not correspond with the mechanisms usually associated with the power law equation.

Recently the creep behavior of single phase alloys has been reanalyzed using the assumption that a threshold stress exists to help correlate the increasing stress exponent and activation energy for creep as stress decreases[37,38]. The threshold stress, σ_0 , is defined as the stress required to initiate dislocation motion[39]. When data for the Ti-50Al alloy were reanalyzed considering the threshold effect, the data fall on a single line,

when temperature compensation is taken into consideration, Figure 21. A similar result was obtained for the other compositions[38]. This result suggests that a single deformation mechanism can explain creep behaviour of single phase γ -TiAl with the use of a threshold stress. No results to date have been published on experimental determination of the magnitude of a threshold stress for single phase γ alloys.

2.4.3.2 Creep of Multiphase Alloys

Considerable creep testing has focused on the multiphase types of microstructures, with particular attention to the fully lamellar structures which exhibit the best creep resistance of near γ -TiAl alloys. The creep properties of near γ -TiAl two phase alloys are often dependent on microstructure, alloy composition, and testing parameters (i.e. stress and temperature). The sections to follow will deal with the dependence of the creep properties on microstructure and stress since these are the most relevant to the thesis work being presented.

2.4.3.3 Dependence of Creep on Microstructure

As mentioned, fully lamellar structures display the best creep resistance when compared to single phase and duplex type microstructures, Figure 22[40]. For lamellar type microstructures several microstructural parameters are often noted in reference to creep behaviour, such as lamellar grain size (LGS), the interlamellar spacing (LS) and the volume fraction of γ grains.

To determine the source of the superior creep resistance for lamellar type microstructures, the effect of grain size on creep behaviour has been examined[20,41]. For a range of grain sizes from 200-1200 μm , the effects of grain size appear to be negligible on the minimum creep rate. This independence of grain size with the minimum creep rate is characteristic of dislocation climb controlled creep processes. However, from Figure 23, the extent of tertiary creep appears to increase as the grain size decreases (comparing the microstructures of Figures 24a to that of b and c). The finer grain size of the microstructure of Figure 24b decreases the length of intergranular cracks that form during the transition to tertiary creep, yielding a lower stress intensity at the crack tip than a coarse grained structure[42]. The higher stress intensity leads to more rapid crack growth and failure with minimal tertiary creep.

The extent of tertiary creep is also dependent on the grain boundary γ grains present in the microstructure of Figure 24a. This earlier transition to tertiary creep is due to the dynamic recrystallization of γ grains along grain boundaries. Examination of the microstructure of Figure 24a after testing revealed a fine network of recrystallized γ grains along the grain boundaries[20]. In contrast to the microstructure of Figure 24c the extended period of tertiary creep is not present due to the absence of the grain boundary γ grains eliminating any dynamic recrystallization from occurring[20]. A similar microstructural result was presented by Es-Souni et al.[43,44] under similar testing conditions where the formation of necklace-like fine γ grains surrounding lamellar colonies were attributed to dynamic recrystallization in a lamellar type microstructure with coarser grain boundary γ present before testing[44]. In the same study [44], a similar microstructure, but with finer grain boundary γ grains was tested and similar creep rates were obtained but lower fracture strains were associated with the finer γ grained structure, consistent with previous results described above[20].

An increase in γ grains resulting in a duplex microstructure leads to a significant decrease in creep properties compared to the fully lamellar structure, as seen previously in Figure 22[40]. Deformed microstructures revealed dynamic recrystallization in the γ grains of the duplex structure with none observed in the fully lamellar structure. A composite type of behavior has been suggested to explain creep properties of varying volume fraction of either γ or lamellar constituent, but this type of behaviour does not agree with the results of Figure 22, where the equiaxed γ would be expected to have the lowest creep resistance.

Results for tests performed on microstructures with increasing volume fraction of lamellar grains between 5 to 80% had virtually no effect on the creep properties at 750°C and 250 MPa, Figure 25[45]. On the contrary, other research has shown that an increase from 55 to 65% lamellar constituent leads to an order of magnitude drop in the minimum creep rate[46]. Reasons for incompatible results may be due to differences in grain sizes of lamellar constituents as the volume fraction of γ grains either increases or decreases, but further testing of low γ grain volume fraction microstructures (of the nearly lamellar type) is required to clarify the effect of γ grains on creep.

Analysis of deformed structures with γ grain constituents by transmission electron microscopy revealed the formation of well developed sub-grain boundaries consisting of arrays of $1/2\langle 110 \rangle$ edge dislocations suggesting glide and climb of dislocations within the γ grains[43]. For the creep curves of Figure 22, the equiaxed γ structure and the γ grains of the duplex microstructure, also showed evidence of sub-grain formation as a result of movement of $1/2\langle 110 \rangle$ type dislocations as well as twinning on $\{111\}$ planes with the density of twins increasing with strain[40]. Analysis of the lamellar constituent in the

duplex structure revealed the presence of dislocation activity, showing deformation occurs within lamellar regions as well[40]. Dislocations were of the $1/2\langle 110 \rangle$ type and movement was confined to individual lamellae with these dislocations frequently being pinned at lamellar interfaces, Figure 26[40]. Pile ups of dislocations were also observed at interfaces and dislocations were bowed out from interfaces[40].

The lamellar interfaces have considerable influence on the deformation characteristics of lamellar type microstructures. The influence of the lamellar interfaces has been linked to two mechanisms[47]. The first is due to the fine interface spacing limiting the glide distance of dislocations therefore reducing dislocation glide[48, 49,50]. The presence of dislocation pile ups at interfaces supports this theory[40]. The second mechanism is that climbing dislocations may become trapped at interface boundaries restricting motion of dislocations and causing bowing of dislocations between interfaces[50]. Bowing of dislocations across interfaces has been observed in creep deformed material[40,50]. The Orowan shear stress, τ_o , required for bowing of the dislocation between interfaces is given as,

$$\tau_o = \frac{Gb}{2\pi L} \cdot \ln \frac{L}{2b} \quad \dots(2)$$

where G is the shear modulus, b is the Burger's vector, and L is the lamellar interface spacing[50]. Referring to the discussion of the single-phase γ alloys where the existence of a back stress was postulated, this same phenomena may be supported by the presence of a shear stress for bowing of dislocations. From the equation above, a finer interface spacing results in a larger shear stress, however experimental evidence to support this is currently lacking for microstructures that are similar, except for interface spacing. There is, therefore, a need to compare directly the influence of lamellar spacing on creep properties of fully lamellar microstructures.

Due to the large grain sizes associated with the lamellar structures there is anisotropy of the lamellar structure, where deformation characteristics depend on the orientation of the lamellae with respect to the stress axis. Grains with lamellae oriented either 0° or 90° to the stress axis, are often referred to as being in the 'hard' orientation, as opposed to grains with lamellae at angles between 30° and 70° being referred to as the 'soft' mode orientation[47]. The properties for single crystals with lamellar grains oriented through 0° to 90° degrees have been examined[51]. From Figures 27a and b, between 30 and 50° (soft mode) tensile strength is at a minimum while the ductility is high, and at 0 and 90° (hard mode) strength is at a maximum while ductility is comparatively lower.

Analysis of deformed crystals oriented in soft mode reveals formation of several $\gamma/\gamma_{\text{twin}}$ interfaces which are parallel to the lamellar direction. As well $1/2\langle 110 \rangle$ dislocation activity is present in other γ lamellae. The occurrence of either mechanism is due to the slight tetragonality of the γ phase, therefore the $\{111\}$ planes are not equivalent, as mentioned in section 2.1, causing deformation by either twinning or $1/2\langle 110 \rangle$ dislocation slip[47]. In hard mode crystals, deformation is characterized by twins in γ lamellae intersecting with α_2 lamellae as well as $1/2\langle 110 \rangle$ dislocation slip. The hard mode is stronger due to the α_2/γ interfaces reducing the distance for dislocation glide and due to the effectiveness of lamellae in preventing twin propagation[52]. To extend the results of this study to a microstructure made up of several lamellar grains may provide information by examining the deformation characteristics of individual lamellar grains as a function of their orientation to the load axis. This may give a more complete understanding of the lamellar structure on creep.

Creep resistance of fully lamellar structures is superior when compared to duplex microstructures, with a part of this creep resistance being attributed to the well interlocked grain boundaries associated with lamellar structures. A serrated type of boundary has been reported to increase creep resistance for austenitic stainless steels [53] and Ni base superalloys[54]. The boundary may serve to restrict grain boundary sliding, and retard crack growth by crack deflection leading to an increased crack length[55]. A study has shown that for a TiAl alloy with interlocked grain boundaries, the creep properties improved as the degree of interlocking increased[55]. The degree of interlocking was controlled by altering the percentage of aluminum in the alloy. In a previous section it was discussed that the interlocking of lamellae can be increased without altering aluminum content, but rather by altering the heat treatment conditions (section 2.3.2). An investigation of the creep properties of similar microstructures with differing grain boundary morphology would help elucidate the role of the serrated grain boundary on creep behaviour.

2.4.3.4 Effect of Stress on Creep

To obtain a relationship between applied stress and the minimum strain rate, stress increment tests are often performed on lamellar type microstructures. By performing such tests, a single sample may be used to generate a complete set of results. This type of test involves increasing the stress, leading to a corresponding increasing strain and strain rate. The results of a typical test are illustrated in Figure 28, where strain rate and stress are compared. Stress exponents for the power-law creep equation, used to help explain the controlling creep mechanism for the single phase microstructure, are also utilized for explaining creep behavior of lamellar microstructures. From Figure 28, the stress

exponent 'n' changes from >7 in the high stress region to ≈ 1.6 in the low stress region, implying a changing creep mechanism, from dislocation creep at high stresses to diffusional creep at low stresses[50]. A similar relationship has been observed by other researchers[42,57]. As previously discussed with the single phase γ microstructure, an exponent of >4.5 implies power-law breakdown (or dislocation glide creep). The existence of an internal back stress, due to the fine interface spacing in lamellar microstructures[42,56] may explain the high stress exponents as with the single-phase alloys described in the previous section.

In stress reduction tests performed to study the creep mechanisms,[50] reductions from high stress levels (from 723 to 586 MPa) shows positive strain immediately after unloading while in the low stress region, there is an elastic contraction followed by a period of zero creep (from 517 to 138 MPa), Figures 29a and b[50]. These results indicate that since an immediate positive creep rate was observed after a stress reduction at high stress, Figure 29a, creep may be glide controlled in this region, while for the lower stress region, creep may be recovery controlled since a zero creep rate was observed, Figure 29b[50]. Similar stress reduction tests performed on other alloys suggest the existence of a back stress opposing dislocation motion[39,58]. The source of this back stress may be the shear stress, τ_o , associated with the bowing of dislocations between glide obstacles. To determine if a back stress exists in lamellar microstructures, stress reduction or decremental tests need to be performed. These decremental test techniques and other creep testing parameters are discussed in the next section.

2.5 Creep Fundamentals

Creep can be defined as the time dependent plastic deformation of materials subjected to a constant load. Depending on the applied load and temperature, creep deformation may lead to failure after a period of minutes or years. Based on the response of many materials, a plot of strain versus time generally leads to creep curves similar to Figure 30, with the three regimes known as primary, secondary and tertiary creep.[59] During the primary stage of creep the metal is hardening and the rate at which the specimen is elongating (the creep strain rate) , $\dot{\epsilon}$, decreases with time. It is assumed that stable substructures develop during this primary creep stage which increase the overall resistance to dislocation motion, hence the decreasing creep rate. During the secondary stage of creep, $\dot{\epsilon}$ is constant and substructures remain unchanged while being strained. This would indicate that the stable substructures developing during primary creep remain unchanged or steady in this secondary region, which is associated with a balance between hardening and softening processes, leading to the term steady-state creep regime being associated with this part of the creep curve[59]. Tertiary creep is characterized as the part of the curve where an increasing $\dot{\epsilon}$ dominates, due to an unbalance in the hardening and softening processes. During this regime several microstructural changes are often occurring, such as microvoid formation, localized necking, and strain hardening of grains leading to dynamic recrystallization all contributing to an increasing $\dot{\epsilon}$ [59].

2.5.1 Steady-state Creep

The secondary creep regime is often characterized as the straight line portion of the total creep curve. As mentioned above, this region has been characterized as having a creep rate that is constant over time, yielding a relationship of the form:

$$\dot{\epsilon}_s = u(\sigma) \cdot v(T) \quad \dots(3)[36]$$

where $u(\sigma)$ represents the dependence of strain rate with stress and $v(T)$ the dependence of strain rate with temperature. By performing creep tests at the same stress level for a group of specimens, and varying the temperature, a straight line relationship is obtained by plotting the logarithm of strain rate with the inverse of temperature, so that $\ln(\dot{\epsilon}_s)$ is proportional to $(1/T)$, Figure 31. This type of temperature dependence is common in many processes including diffusion and oxidation[36], which obey the familiar Arrhenius equation,

$$\dot{\epsilon}_s \propto \exp\left(\frac{-Q_c}{RT}\right) \quad \dots(4)$$

This shows that the secondary creep rate increases exponentially with temperature. The slope of Figure 31[36] is equal to $-Q_c/R$ where Q_c is activation energy for creep and R is the universal gas constant. It is often found that the values of Q_c are close to that for the activation energy for lattice self-diffusion, Q_{sd} , thus emphasizing the importance of diffusion during creep at high temperatures, above 0.4 of the absolute melting temperature (T_m) of the metal, where atomic movement is greatly enhanced by diffusion[36].

The steady-state strain rate is also a function of the stress, and in a similar manner as was described for the temperature dependence, creep tests can be performed at the same temperature for a group of specimens but at various stress levels for each specimen. A straight line relationship is often observed, Figure 32, when the $\log(\dot{\epsilon}_s)$ is plotted versus $\log(\sigma)$, this suggests a relation for steady-state creep and stress as,

$$\dot{\epsilon}_s \propto \sigma^n \quad \dots(5)$$

where the exponent n is the gradient of the plot in Figure 32.

By combining equations (4) and (5) for the temperature and stress dependence respectively, on the steady state strain rate, the familiar power law equation is described,

$$\dot{\epsilon}_s = A\sigma^n \exp\left(\frac{-Q_c}{RT}\right) \quad \dots(6)$$

where A is a constant dependent on microstructural factors. The values of both n and Q_c vary in different stress and temperature regions.

For test temperatures of around $0.7T_m$ and above with low stresses applied, $Q_c \approx Q_{sd}$ and $n = 1$. At high stresses Q_c is still approximately equal to Q_{sd} but n varies from 4-6. For lower test temperatures of around $0.4T_m$, $Q_c \approx 0.5Q_{sd}$ and $n = 1$, for low stresses, while for higher stresses Q_c remains unchanged at $0.5Q_{sd}$ but n ranges from 4-6. These transitions for n and Q_c are usually explained by the creep behaviour being governed by a different mechanism of creep. The two mechanisms often associated with creep deal with diffusional and dislocation processes.

2.5.2 Diffusional Mechanisms of Creep

The activation energy for diffusion is dependent on the energy of the bonds between the atoms diffusing and the neighbouring atoms. This means that surface atoms and atoms located at grain boundaries (g.b.'s) have lower activation energies since they have fewer neighbours to impede their motion. At high temperatures when lattice diffusion is rapid, the diffusion along grain boundaries and surfaces is negligible compared to the overall diffusion, since the surface areas and disordered g.b.'s are minimal when compared to the volume of the whole specimen. On the contrary, as temperature decreases these easy diffusion paths (g.b.'s and surfaces) become preferred for diffusion and account for most of the diffusion in the sample.[36] A creep theory

developed by Nabarro and Herring explains the time dependent strain, solely by diffusional transport of atoms. At high temperatures Nabarro proposed that under a stress field, vacancies are generated at tensile stress boundaries and these diffuse to compressive stress boundaries. This results in matter moving in the opposite sense and hence extension or straining of the sample. The steady-state strain rate for the Nabarro-Herring [59] relation is given as,

$$\dot{\epsilon}_s = C \frac{D_{SD} \Omega \sigma}{d^2 k T} \quad \dots(7)$$

where D_{SD} is the diffusion coefficient for lattice self diffusion, Ω is the volume of a vacancy, k is Boltzman's constant, d is the average grain diameter and C is a constant approximately equal to 10. This equation is consistent with the previous equation derived for power law creep with an exponent $n=1$ and $Q_c=Q_{SD}$ where $D_{SD}=D_0 \exp(-Q_{SD}/RT)$ for lattice self diffusion[36].

The diffusion creep theory proposed by Nabarro and Herring only considered diffusion through the lattice. Atoms can also diffuse by grain boundary diffusion. The diffusion of atoms at grain boundaries is easier since fewer atoms impede motion. By adding the effect of grain boundaries the Nabarro-Herring relation is modified to yield that described by Coble [59] as,

$$\dot{\epsilon}_s = C \frac{D_{GB}}{d^2} \cdot \frac{\delta}{d} \cdot \frac{\Omega \sigma}{k T} = C \frac{D_{GB} \delta \Omega \sigma}{d^3 k T} \quad \dots(8)$$

where δ is the grain boundary width and δ/d represents the area of grain boundary zone intersecting a unit area of a crystal with average grain diameter d . The value D_{GB} is the diffusion coefficient for grain boundary self-diffusion. Here the dependence of the relation on grain size is noted as more grain boundaries are available for assisting diffusion in a fine grained sample. Comparison with the power law creep equation, equation (7) indicates that Coble creep corresponds to a stress exponent of $n=1$ and the

activation energy for creep of $Q_c < Q_{SD}$, since lattice diffusion becomes more difficult as temperature decreases towards around $0.4T_m$. Therefore Coble creep may be the dominant mechanism for low stress and low temperature tests around $0.4T_m$.

2.5.3 Dislocation Creep Mechanisms

When the diffusional creep models described in the previous section are applied to intermediate temperature and higher stresses and compared to real data, the strain rate is grossly under estimated. For example for copper tested at 580°C and 25 MPa, the strain rate was 10^{-5} s^{-1} while the Nabarro-Herring and Coble creep models predicted 0.35×10^{-11} and 0.13×10^{-12} respectively[36]. There is therefore a need for another theory to explain creep deformation at higher stress levels.

Several dislocation theories exist for creep deformation which focus on the work hardening and recovery principles often used to describe the secondary creep regime, as mentioned in section 2.5. When a metal is deformed it develops dislocations which glide and may pile up at obstacles, Figure 33. This represents the hardening process. These dislocations may climb over the obstacle and begin to glide again in a new slip plane, and the dislocation source may emit another dislocation allowing creep to continue. Dislocation climb is a new degree of freedom available to edge dislocations confined to slip in their own glide plane. Through the climb mechanism they are able to overcome energy barriers and continue gliding on another slip plane. This climbing of dislocations represents the recovery process[36]. Equation 9 relates the hardening (h) and recovery (r) processes to steady-state strain rate

$$\frac{d\epsilon}{dt} = \dot{\epsilon}_r = \frac{r}{h} \quad (9)$$

This indicates that steady state during creep is reached when the rate of work hardening is balanced by the rate of recovery. Relations defining the steady-state creep rate for dislocation theories generally lead to stress exponents around 4 with expressions for $\dot{\epsilon}_s$ yielding,

$$\dot{\epsilon}_s = \frac{r}{h} \propto v \sigma^4 \quad \dots(10)$$

where v is the mobility of climbing dislocations and is dependent on temperature and may be described with an Arrhenius type of relation for activation energy. This leads to a power law dependence for dislocation controlled creep as in Equation (6), repeated here for clarity,

$$\dot{\epsilon}_s = A \sigma^n \exp\left(\frac{-Q_c}{RT}\right).$$

2.5.3.1 Back Stress

Another factor that must be accounted for by dislocation creep theories is the existence of a threshold or back stress resulting from the interaction of moving dislocations with substructures, as described earlier in sections 2.4.3.1 and 2.4.3.4. An often used model [36,39] utilizes the work hardening recovery theory. It involves growth of three dimensional arrays of dislocations that form within a grain interior, known as sub-grain boundaries which form the substructure. In this model the idea that deformation strengthening comes from the refinement of the sub-grain structure of dislocations and recovery from the coarsening of the sub-grain structure by diffusion controlled mechanisms such as climb. This theory explains work hardening resulting

from the increase in dislocation density and creates a finer mesh of sub-grain boundaries. Experimental observations show incubation periods (Δt) after small stress reductions ($\Delta \sigma$). This delay or incubation period is taken as the time required for the network to grow to its new steady-state size. Once this has occurred, the reduced stress level is able to break dislocations free from their pinning points[58].

The back stress associated with this theory is measured by performing a number of stress reductions after a period of steady state creep is measured. Approximately 5% of the applied stress is removed at each decrement after which an incubation period is recorded. A series of incubation periods is recorded as in Figure 34a, and with decreasing stress the delay times become increasingly longer. By plotting the cumulative incubation periods versus the cumulative stress reductions, the back stress is determined as the asymptotic value of the remaining stress where the incubation period reaches an infinite value, Figure 34b[58]. Using this threshold or back stress concept, the idea of a net stress $\sigma - \sigma_0$ is used to define the creep rate with the power-law creep equation as,

$$\dot{\epsilon}_s = A(\sigma - \sigma_0)^n \exp\left(\frac{-Q}{RT}\right) \quad \dots(11)$$

Using this relationship in terms of a net stress rather than an applied stress, activation energies approach those for lattice self diffusion and stress exponents are reduced to 3.5 to 4.

2.5.4 Creep Testing

For operation of components at elevated temperatures the design criteria must provide for no excessive distortion of the component over long term operation and creep

rupture should not occur during operation of the component. Although the stresses encountered during the actual service of a component are complex non-steady state and multiaxial stresses, creep tests are most often performed in a uniaxial manner under one temperature condition. Most component design tests and fundamental creep studies are done using tensile creep or stress rupture equipment. Short term tests of around 100 hours, are done for theoretical studies of fundamental creep behaviour of a particular material. In general, long term test programs are required to generate design data, but some short tests are done for quality control for specific creep strength specifications to be met.

Tensile creep testing is the most frequently used test method for obtaining creep data. The basic schematic of such a tensile loading creep frame is shown in Figure 35. This type of machine is only useful for stress rupture testing where time to failure, t_f , and final elongation of the sample, ϵ_f , are measured since no measurement of strain rate is available with such a configuration. For the measurement of strain rate the same machine may be used but an extensometer must be attached to the specimen with grooves machined into the specimens which can attach to knife edges in the extensometer assembly, Figure 36. The time dependent elongation of the sample can then be measured by using a linear voltage displacement transducer (LVDT) which uses a voltage to determine the elongation from movement of the extensometer. In this way, both the time dependent deformation of the specimen can be recorded as well as t_f and ϵ_f . A complete description of the components comprising a creep testing system will be described in a later section.

2.6 Summary

From the review of the current literature pertaining to near γ -TiAl, the role of microstructure on creep properties has been reviewed. From this review several areas of knowledge require greater fundamental understanding to determine the effects of individual microstructural constituents on the overall creep behaviour of lamellar type microstructures. Firstly, the volume fraction of γ grains has been examined by other researchers but with contradictory results[45,46]. This microstructural parameter may be useful since a tradeoff in creep resistance for an increase in ductility may be achieved with a nearly lamellar type of microstructure, which may exhibit reasonable creep resistance and better ductility than fully lamellar microstructures. Secondly, the interlamellar spacing may be the major source of strengthening in the fully lamellar microstructures. Similar to particle strengthened materials which gain strength from an increased Orowan bowing stress required for dislocation motion, lamellae may act as similar obstacles to dislocation motion. As well the orientation of individual lamellar grains may help explain the deformation characteristics observed in lamellar grains. Thirdly, the often serrated grain boundaries of lamellar microstructures may also effect the creep properties. As mentioned serrated grain boundaries have been known to effect the creep resistance of other materials[53,54]. Lastly, the effect of stress on creep properties was reviewed and the idea of a back stress discussed. The presence of a back or threshold stress may help explain the high stress exponents often associated with lamellar microstructures.

It is the objective of this thesis work to investigate the dependence of creep properties on these various microstructural parameters. In this way, this thesis work will

contribute to an enhanced fundamental understanding of creep of near γ -TiAl alloys and lead to an improved capability for alloy and microstructural design of γ -TiAl alloys.

CHAPTER 3

3. Specific Objectives and Methodology of Thesis

1. The first objective of the thesis, as mentioned at the end of the previous section, is to determine the influence on the creep behaviour of varying the γ grain volume fraction. Previous research has compared creep properties with microstructures with a variation in γ grain volume fractions but results have been inconsistent[45,46]. This nearly lamellar type of microstructure should yield a good combination of creep and ductility, which is lacking in fully lamellar structures.
2. Secondly, the influence of lamellar spacing on the creep properties is to be examined. Currently there is a lack of information available on the effects of lamellar spacing on creep properties. Since this is the major microstructural constituent in most structures, and may be the source of improved creep resistance, data are required to help elucidate the role of lamellar interface spacing on creep behaviour.
3. Thirdly, associated with fully lamellar structures is a changing grain boundary morphology. Serrated or planar grain boundaries may be a source of strength or weakness in lamellar structures and thus experimental evidence is required to help support or refute their influence on creep.

To accomplish these objectives the thesis work consists of several steps.

- A) The development of controlled heat treatments that systematically vary individual microstructural parameters.
- B) Characterization of microstructures prior to creep testing through quantitative optical microscopy and transmission electron microscopy.

- C) Creep testing for determination of properties using monotonic creep tests. Incremental stress tests and the consecutive stress reduction method will be performed.
- D) Characterization of creep deformed microstructures using optical and transmission electron microscopy.

This work is aimed at developing improved guidelines for the microstructural design of near γ -TiAl alloys for creep resistance, but also, to develop a mechanistic understanding of the creep deformation of γ -TiAl alloys with a lamellar microstructure. Only through a mechanistic understanding can the risks associated with the application of near γ -TiAl alloys in the envisaged critical applications be appropriately determined.

CHAPTER 4

4.0 Materials and Experimental Procedure

4.1 Material Preparation

The material used for this thesis was prepared from metal powders consolidated through hot isostatic pressing (HIP'ing). Through the use of powder metallurgical techniques, the difficulties associated with fabrication of near γ -TiAl can be minimized since fully dense shapes are produced which only require machining.

Powder, obtained from Crucible Compaction Metals, was produced by the titanium gas atomization process (TGA) as depicted in Figure 37. In the TGA process a 27 kg charge is melted in a cold wall induction crucible. By utilizing a cold walled crucible, a thin skull of the charge lines the crucible helping to promote cleanliness without the need of a refractory lining which often causes contamination[60]. The molten alloy is then atomized by pouring into a non-ceramic tundish with a refractory metal nozzle at the bottom which produces a stream of the molten alloy. This stream is atomized and solidified by a high pressure jet of argon gas, carrying the powder into a cyclone from the atomizer tower. It is then collected in a canister. The powder is typically screened to -35 mesh (500 μ m) and processed through a magnetic separator to remove particles magnetized during atomization. Interstitial content after the TGA process for TiAl intermetallics is 800 ppm oxygen, 200 ppm carbon, 50 ppm nitrogen, and 20 ppm hydrogen. The powder utilized for this research had the chemical composition of Ti-48.1 at% Al.

To consolidate the powder it must first be hermetically sealed within a metal tube. The tubes used for canning of the powder were made from commercial purity titanium tubing with a 15 mm diameter and 1 mm thick wall. Sections of tube were cut to 35 cm lengths and one end of the tube was hydraulically crimped and sealed by tungsten inert gas welding. The tube end to be crimped was first heated with an acetylene torch to anneal the titanium and prevent cracking prior to crimping. The tubes were then thoroughly cleaned with petroleum ether and checked for leakage on the vacuum system, Figure 38. A vacuum of $<5 \times 10^{-5}$ mbar is attained with a properly sealed end. Following leakage testing the tubes are filled with the intermetallic powder inside a glove box with an argon atmosphere to prevent contamination of the powder with air, Figure 39. The cans are filled to yield 18 cm lengths of TiAl powder. For consolidation to occur the tube filled with powder must be sealed under a vacuum. Again the tube is placed on the vacuum system and a vacuum of $<5 \times 10^{-5}$ mbar achieved. While under a vacuum the tubes are placed in a furnace at 500°C for out gassing of the powders for a 24 hour period. Following cooling, the tubes are sealed again by hydraulically crimping the unsealed end after annealing with an acetylene torch, followed by welding. Figure 40 illustrates a tube being hydraulically crimped. The powder filled tubes may now be hot isostatically pressed to yield solid rods of TiAl.

4.2 Hot Isostatic Pressing

Hot isostatic pressing subjects powder to high temperatures and pressures in an autoclave system pressurized with argon gas. Pressure is exerted on all sides of the object equally with the range typically between 20 to 300 MPa, while the temperature is usually within the limits of 480°C for aluminum and up to 1700°C for tungsten. There are

several parameters which must be addressed when implementing a HIP process, including:

- 1) temperature and pressure cycle to yield a fully consolidated object,
- 2) resulting microstructure after the HIP'ing process, and
- 3) resulting shape of the object after HIP'ing.

As well as the actual HIP'ing parameters having an effect on the consolidation, the powder properties also have an effect on the quality. Important properties include particle shape, mean particle size, particle size distribution, composition and impurity content[61]. If powder quality and HIP'ing parameters are optimum, the end result of the HIP'ing process is a fully consolidated object with a near-net shape. As well as providing the ability to heal porosity in metal parts, HIP'ing can also provide a part that is ideally suited for a particular application due to the microstructural control available during HIP'ing.

The hermetically sealed powders of near γ -TiAl were HIP'ed in an Autoclave Engineers press, shown schematically in Figure 41. The unit is capable of operating at 2000°C and 207 MPa with a specimen chamber of 240 mm by 120 mm. Once the vacuum-encapsulated powder is loaded into the press, it is purged with argon three times to ensure all air is removed. The pressure is then set at 55 MPa with the remaining pressure generated by the heating of the unit. For the material used in this thesis the HIP conditions were 1250°C for 2 hours at 200 MPa to achieve full consolidation. A schematic of the HIP cycle is shown in Figure 42.

4.3 Heat Treatments

Following HIP'ing a fully consolidated tube is obtained. The ends of the tube are cut off and the outer titanium casing machined away to expose the TiAl rod. These rods are cut into various lengths depending on the use of the material, i.e. heat treatment or creep blanks. Heat treatment blanks are cut into 1 cm long pieces, wrapped in tantalum foil and sealed inside quartz tubes back filled with argon. The tantalum prevents the silicon in the quartz from diffusing into the TiAl specimen. The quartz tube with the protective Ar atmosphere is required due to the high heat treatment temperatures and prevents severe oxidation of the specimens. The specimens to be used as creep blanks are cut to at least 7 cm lengths and a similar procedure as described above is used to prepare the specimens for heat treatment. Figure 43a to g depicts the complete cycle from TiAl powder to a machined creep specimen.

Heat treatments were performed in a Lindberg furnace with a maximum temperature 1500°C. The furnace was controlled by a Microstar furnace controller. A type S platinum/rhodium thermocouple was used to constantly monitor the temperature during heat treatments. Specimens were heat treated between 1350°C and 1400°C for durations varying from 30 minutes to 2 hours, followed by air or furnace cooling. A stabilization heat treatment of 950°C for 48 hours was applied to prepare the microstructure for long time exposure at temperature during creep testing.

4.4 Metallography

To prepare specimens for optical microscopy both electrolytic and mechanical polishing procedures were used. Electrolytic polishing was performed on heat treatment

specimens for microstructural analysis while mechanical polishing was used for creep deformed samples. An effect associated with electrolytic polishing is the formation of polishing pits. Due to this effect creep deformed specimens were mechanically polished to minimize extraneous porosity which may obscure cracking or void formation as a result of creep deformation.

4.4.1 Optical Microscopy Preparation

Electrolytic polishing requires the specimen to be ground following heat treatment on 240, 320, 400, 600 and finally 800 grit silicon carbide paper prior to polishing. The electrolyte consists of methanol, ethylene glycol, and perchloric acid in a 10:6:1 ratio by volume. The electrolyte is cooled to between -25 to -30°C with liquid nitrogen. With an applied voltage of 30V, the specimen which is an electrode, is immersed in the electrolyte for approximately 4 minutes. Following electrolytic polishing the specimen may be etched for optical examination.

The etchant used consists of 25 ml of distilled water, 50 ml of glycerol, 25 ml nitric acid and 2 ml hydrofluoric acid. The etchant is applied to the specimen for approximately 10 seconds or until the surface loses its luster. The specimen is then rinsed with water to stop the etching process, and following thorough ultrasonic cleaning with methanol the specimen may be viewed under the microscope.

Mechanical polishing requires similar grinding as in the electrolytic preparation with grinding up to 600 grit paper followed by 600 grit cloth and then the 800 grit SiC paper. Following grinding, the specimen is thoroughly cleaned and then polished on

successive 9, 3, and 1 μm diamond wheels until a mirror finish is obtained. Once the finish is appropriate the sample is etched with the same procedure as described above.

Optical Microscopy was done on a Olmypus PMG3 microscope with Buehler Omnimet image analysis software used to determine γ grain volume fractions.

4.4.2 Transmission Electron Microscopy Preparation

Foils for transmission electron microscopy (TEM) were prepared from both heat treated and creep deformed specimens. Using a diamond cut-off wheel, heat treated specimens were sectioned to 0.65 mm thick disks. The thin disks were then ground on SiC paper up to 600 grit as in the procedure for preparing mechanically polished specimens for optical examination. Each side of the disk was ground until the specimen was between 80-100 μm thick. A punch is used to remove 3 mm diameter disks from the larger ground disk. For creep deformed specimens, the gauge length was machined to 3 mm diameter before specimens were sectioned from it. A sample must be 3 mm in diameter to be accommodated in the TEM goniometer/sample holder.

Once specimens are ground they are jet polished in an electrolyte consisting of 64% methanol, 34% butanol-1 and 5% perchloric acid. Conditions for obtaining optimum TEM foils requires a temperature between -48 and -50°C. The settings on the Tenupol-3 jet polish apparatus were: an applied potential of 12 V, a flowrate of 5.5, a current setting of 0.5A, and photosensitivity of 0.5-0.75.

All TEM examination was undertaken using the Philips EM420 of CURFEM (Carleton University Research Facility for Electron Microscopy) operated at 120 kV. To determine the interlamellar spacing, foils were oriented so that lamellar interface planes were parallel to the incident electron beam. Multiple foils were examined with this technique and the average interface spacing was determined from bright field images of the foils. Deformed specimens were prepared from the gauge length of creep specimens, with foils cut normal to the stress axis, so that angle of tilt between the stress axis and the plane of the lamellar interfaces of individual lamellar grains can be determined from the goniometer tilt angle when lamellar interfaces are parallel to the incident electron beam. The measurable angle of tilt was limited to 60° due to goniometer tilting limitations. Due to foil distortion from jet polishing, the lamellar orientation is determined to within $\pm 3^\circ$.

4.5 Creep Testing

Creep tests were performed on a Satec creep frame equipped with a set of extensometer rods for strain measurement. A typical frame is illustrated in Figure 44. This type of frame is of the constant load type described in section 2.5.4. The frame operates with a 3:1 or 20:1 lever arm ratio depending on the stress level or accuracy required. Specimen elongation was measured with linear voltage displacement transducer (LVDT) attached to the extensometer assembly. Prior to testing the LVDT was calibrated and the output recorded and plotted to determine the linear operating range of the LVDT, Figure 45.

A three zone furnace provided heating of the specimen at 760°C for all tests with maximum temperature drift of $\pm 2^\circ\text{C}$, in accordance with ASTM standards. The

temperature was monitored by a type K thermocouple attached to the center of the specimen. A Fluke 220B data logger was used to record temperature and voltage from the LVDT with output provided on a strip chart.

Stress increment testing required periodically increasing the stress by increasing the load. This was done simply by the addition of supplementary weights on the weight pan. The stress level was corrected from the measured strain value for a more accurate stress level determination particularly at high strain levels.

Stress decrement testing required removal of some of the load (5% decrements) to obtain a lower stress level. This was accomplished by stacking of weights in predetermined groups for easy removal with minimum movement of the weight pan. Following removal of a load the voltage was monitored with a PC based data acquisition system providing measurement of data every second. Incubation periods were determined from these data as periods of zero strain rate (i.e. constant voltage).

Prior to every test, specimen dimensions were measured on which stress calculations were based. Care was taken in performing every test and no tests were carried out without all conditions met, such as leveling of the lever arm, accurate load calculation, proper specimen placement within the knife edge grips, and recalibration of LVDT.

Multiple tests were not performed on microstructures since good test repeatability is achieved when attention to experimental procedure is respected. Figure 46 illustrates 2 creep curves for the same microstructure tested.

CHAPTER 5

5. Effect of γ Grain Volume Fraction on Creep Behaviour

Using the method described in section 4.3 for heat treating of specimens, the resulting microstructures developed to obtain a variation in the volume fraction of γ grains are described in this section. The microstructure prior to heat treatment is first discussed followed by the developed microstructures. Creep testing results on the various microstructures are presented and a discussion follows after each section.

5.1 Results of Heat Treatments for Variation in γ Grain Volume Fraction

Following HIP'ing, the microstructure obtained was composed of γ and α_2 grains, with the γ grains in an equiaxed form with a grain size of approximately 15-20 μm , as illustrated in Figure 48. The α_2 phase in Figure 48, has a lighter contrast and with a much lower volume fraction. Within several of the γ grains, several annealing twins cross the γ grains. Another feature that was noted in the as HIP'ed microstructure was the evidence of the prior dendritic structure often found in the P/M produced microstructure, Figure 49. A complete discussion on the evolution of the as HIP'ed microstructure is available in ref.[63]. The as HIP'ed microstructure is the starting microstructure for all heat treatments described below.

As mentioned a series of heat treatments was undertaken to develop microstructures with a range of volume fractions of γ grains. By heat treating in the upper region of the $\alpha+\gamma$ phase field at 1350°C, Figure 47, the as HIP'ed microstructure consisting of α_2 and γ grains, transforms to a duplex (DP) type of microstructure composed primarily of fine γ and lamellar grains, Figure 50a. The lamellar grains result from transformation of a portion of the γ grains to α , which on cooling form the lamellar

structure. This type of transformation sequence was described in section 2.2.2.2. Upon closer examination of the microstructure of Figure 50a, the presence of incompletely transformed lamellar grains become evident. Formations of lamellar plates appear to nucleate at grain boundaries. This is expected due to the disorder offered at the boundary and the subsequent lower energy for formation. There are also some γ grains with α_2 plates traversing the grain often with different orientations, Figure 50a, this may be the result of a third formation mechanism for the lamellar morphology (in addition to the two mechanisms presented in section 2.2.2.1). This third mechanism, involves the nucleation of α plates out of a γ matrix, with the subsequent ordering of the α to α_2 . This reaction occurs during heat treatment of two phase alloys in the $\alpha+\gamma$ region of the phase diagram. The crystal structure of the γ phase permits the nucleation of the α phase on four possible planes leading to four orientations within a grain, unlike the $\alpha \rightarrow \gamma$ reaction which has only one habit plane leading to the $(0001)//\{111\}$ relationship observed in the first two mechanisms. The resulting microstructure consists of alternating α_2 and γ plates with the usual crystallographic orientations but with the γ plates having the same crystallographic orientations within the grain[15].

Grain sizes were estimated for the duplex microstructure as $\sim 35 \mu\text{m}$ for the γ grains and an average lamellar grain size of approximately $60 \mu\text{m}$. The volume fraction of γ grains was determined to be between 80-85% γ . A range was determined due to the difficulty associated in resolving lamellar grains from γ grains due to the poor contrast of the images.

By increasing the heat treatment temperature to 1365°C for 1 hour, within the $\alpha+\gamma$ phase field, the microstructure transforms to the nearly lamellar (NL) type also discussed in section 2.2.2.2. This microstructure also consists of lamellar and γ type grains. The volume fraction of γ grains decreases compared to the duplex microstructure, since upon

cooling more lamellar grains are formed from the increased presence of transformed α , consistent with the phase diagram, leading to the nearly lamellar microstructure of Figure 50b. An increase in lamellar grain size from 65 to 90 μm is noted from a comparison of nearly lamellar and duplex microstructures with a corresponding increasing the equiaxed γ grain size from 30 to 35 μm . The volume fraction of γ grains was determined to be between 20-25%. Also, γ grains appear primarily around lamellar grain boundaries.

The α transus for this alloy is 1370°C [19] and heat treatment at or just above the α transus at 1370°C and 1375°C for 1 hour does not allow sufficient time for complete transformation from γ to α , resulting in the microstructures of Figures 50c and d, composed of γ and L grains, again these are referred to as NL. The volume fraction of γ grains for each of these microstructures was determined as 15-20% and 7-10% respectively. By heat treating above the α transus, a further increase in lamellar grain size occurs coupled with a decrease in γ grain size as the temperature is increased. Again, this is due to increased transformation of γ to α . The transformations associated with this type of microstructure was covered in section 2.2.2.2.

Heat treating sufficiently above the α transus, 1380°C for 1 hour results in complete transformation of $\gamma \rightarrow \alpha$ and upon cooling a fully lamellar (FL) microstructure is formed with almost no γ grains remaining, Figure 50e. The development of this type of structure was described in section 2.2.2.1 with further details available elsewhere[8]. Complete microstructural details are provided in Table 2 for the microstructures of Figure 50 a to e.

5.2 Results of Creep Tests on Microstructures with Variation in γ Grain Volume Fraction

Results from creep tests for the effect of γ grains on creep properties are compared in Figure 51 as percent engineering strain versus time in hours. The results show increasing creep resistance as the microstructure changes from the DP to NL to FL illustrated in Figures 50 a to e. (It is noted that at the high strain levels of the DP condition there is an error associated with engineering strain.) This increasing creep resistance corresponds to a decrease in the γ volume fraction and an increasing lamellar grain size (seen from the parameters of Table 2). There is a large reduction in the creep life with only a small volume fraction of γ grain present as seen in Figure 52a, where the NL (20-25% γ grains) and DP show little difference in creep life. In contrast, the minimum strain rate is less affected by the presence of a small volume fraction of γ grains as seen in Figure 52b. (Minimum strain rate is less affected by engineering strain measurement errors, since the slope of each curve was used to measure the strain rate prior to tertiary creep.) As well, from a comparison of the strain rate versus the strain for the NL microstructure with 7-10% γ grains and the FL microstructures, Figure 53, there is only a minimal difference in the minimum strain rate. A larger difference is observed with the steady increase of strain rate with strain for the NL microstructure. These results indicate that tertiary creep occurs earlier and to a greater extent as the γ volume fraction increases.

5.3 Deformed Microstructures

Analysis of deformed microstructures revealed that γ grains exhibit greater deformation compared to lamellar grains for a nearly lamellar structure with 20-25% γ grains), Figures 54a and b. In Figure 54b, strain appears to be accommodated by the γ

grains that surround the lamellar grains which remain relatively undeformed. As well γ grain deformation occurs in an increasing larger volume of the specimen as the amount of γ grains increases, consistent with the increasing fracture strain. Formation of fractures for NL microstructures with volume fractions between 15-25% γ occurs primarily at lamellar/ γ grain boundaries after considerable γ grain deformation, Figures 55a and b. For the NL(7-10% γ grains) and FL microstructures with low volume fractions of γ grains the fracture is brittle and intergranular as seen in Figures 56a and b.

Comparison of TEM microstructures before and after testing indicate an increasing density of dislocations within the γ grains and lamellar grains of the DP (80-85% γ) structure, Figures 57a to c. This result is consistent with that described in section 2.4.3.3 where dislocations were observed in both lamellar and γ grain regions[40]. Another observation was the presence of several γ grains, observed from the deformed creep specimen, that appear to be free of dislocations, Figure 58a and b.

Similar results were obtained for the NL structure of Figure 50c. Creep deformed structures were observed with increased dislocation density within lamellar and γ grains, Figures 59a and b. There is also evidence of dislocation free grains in this microstructure after testing, Figure 60.

5.4 Discussion of Creep for Microstructures with a Variation of γ Volume Fraction

The results of Figure 51 demonstrate that microstructure has a large influence on the creep properties of near γ -TiAl. From the data on Table 2 and Figure 51, the microstructures vary in terms of γ volume fraction and lamellar grain size. Previous results have indicated[20] that lamellar grain size does not influence the minimum creep rate. This implies that the volume fraction of γ grains is the controlling microstructural

feature governing the creep properties. This agrees with results indicating deformation of lamellar grains is minimal compared to the bulk of deformation occurring in γ grains, Figure 54b. Several factors may contribute to the increased deformation of the γ grains. Firstly, grain boundary sliding (GBS) may occur more easily between the γ grains surrounding the lamellar grains, Figure 54a. Due to the finer γ grains, GBS is accelerated because of the increased grain boundary area associated with the finer grain size. An analogous GBS phenomenon has been cited previously during creep of similar lamellar microstructures[45,64] and during hot deformation of Ti-alloys[65]. Secondly, the preferential deformation of the γ grains causes localized dynamic recrystallization (DRX) of the γ grains. This reduces the γ grain size, further increasing the tendency for GBS and thereby accelerating the creep rate causing early onset of tertiary creep and rapid fracture. As reviewed in section 2.4.3.3, this phenomenon has been found by other researchers for similar microstructural conditions[20,43,44]. As well, a similar relationship between DRX and creep properties has been observed in other near γ -TiAl alloys with nearly lamellar microstructures[57].

From TEM results on the DP and NL (15-20% γ grains) microstructures, there are several small possible dislocation free grains, which may be the result of dynamic recrystallization, as shown in Figures 58a and b and Figure 60. During dynamic recrystallization, dislocations rearrange themselves into sub-grain boundaries of lower energy configurations creating a network of finer grains. Therefore, increasing the volume fraction of γ grains increases GBS and DRX. This series of creep tests, indicate that for NL microstructures the creep properties are controlled by the γ grains that surround the lamellar grains.

CHAPTER 6

6.0 Effect of Lamellar Interface Spacing on Creep Behaviour

Using the heat treatment method described in section 4.3 two microstructures were developed to determine the effect of lamellar interface spacing on creep properties. To obtain easily reproducible microstructures, air and furnace cooling (AC, FC) were employed.

6.1 Results of Heat Treatments for Differing Lamellar Interface Spacing

Two microstructures were developed for creep testing using air and furnace cooling to control the interface spacing. The air cooled microstructure developed for testing of lamellar spacing effect was the same as previously described as the fully lamellar structure tested for the effect of gamma grains on creep properties, which was heat treated at 1380°C for 1 hour, Figure 50e. The furnace cooled microstructure was heat treated at 1400°C for 1/2 hour, and is shown in Figure 50f. Time and temperature were selected to obtain similar grain sizes for both microstructures. Although heat treated for a longer period of time, the air cooled microstructure has a smaller grain size due to the lower temperature providing less energy to drive grain growth (see section 2.2.2.1). The grain sizes determined for these microstructures were 290 and 325 μm for the air and furnace cooled conditions respectively. To determine an estimate of the average lamellar interface spacing, TEM foils were made of undeformed heat treatment samples, and with the procedure outlined in section 4.4.2, the average lamellar spacing for air and furnace cooled microstructures was determined to be 120 and 450 nm respectively, Figures 61a and b. An important observation from several foils was the low dislocation density observed for both air and furnace cooled specimens. The grain boundaries of the furnace cooled microstructure (Figure 50f) show a greater degree of lamellar interlocking in contrast to the relatively planar grain boundaries of the air cooled sample (Figure 50e).

6.1.1 Discussion of Microstructural Development

As discussed in section 2.3 dealing with the morphology of lamellar microstructures, the lamellar spacing is dependent on the cooling rate. For air cooling the increased nucleation rate, but lower rate of diffusion leads to thin plates with narrow spacing. Furnace cooling leads to a lower nucleation rate but higher diffusion due to the higher temperature for a longer time and therefore thicker plates with a wider spacing, Figure 61a and b.

In addition to the lamellar spacing, the cooling rate also effects the grain boundary morphology. Grain boundaries in the furnace cooled microstructure exhibit more interlocking relative to the planar grain boundaries of the air cooled sample, Figures 50e and f. Although the development of the interlocking morphology of the furnace cooled structure is not completely understood, section 2.3.2 describes the mechanism of discontinuous coarsening which accounts for the thickening of the plates at grain boundaries by the migration of γ plates into adjacent grains. The serrated grain boundaries may be an early stage of discontinuous coarsening where γ plates have only begun migrating and thickening into the adjacent grain. Although not part of this work it has been shown [19] that varying the heat treatment time by performing long time heat treatments may also alter the morphology of the grain boundaries, with longer heat treatment times yielding a more serrated boundary (see section 2.3.2).

6.2 Results of Creep Tests Performed on Microstructures with Differing Lamellar Spacing

The creep tests performed on the two microstructures with differing lamellar spacing included monotonic tests to fracture, stress increment tests and stress decrement tests. Results from monotonic tests to fracture are illustrated in Figure 62. From these

results it is evident that creep properties are greatly influenced by the rate of cooling applied to a specimen following heat treatment from within the α phase field. A comparison of the data of Figure 62 reveals a higher strain rate, shorter creep life and greater overall strain at fracture for the furnace cooled specimen, Table 2. The air cooled specimen failed at $\approx 6.5\%$ strain while the furnace cooled failed at $\approx 18\%$. The creep life of the air cooled microstructure is doubled and the strain rate is decreased by a factor of ≈ 5 , compared to the furnace cooled structure. In addition a comparison of the strain rate versus strain shows no steady state creep rate but rather a minimum rate for the furnace cooled specimen similar to previous results for specimens with high γ volume fractions, Figure 63. In addition to the tests to failure, an additional test was performed on the furnace cooled microstructure, but the test was stopped at 6.5% strain, the approximate failure strain of the air cooled specimen.

6.2.1 Incremental Tests

A portion of the creep curve for the furnace-cooled microstructure is provided in Figure 64 showing the response to a stress increment and the corresponding increase in creep rate. The creep strain rates for each stress tested for the fully lamellar microstructures are presented in Figure 65. Included in Figure 65 are the results of the monotonic tests discussed previously, with the similarity in strain rates at comparable stress levels noted. This result verifies that under the testing conditions used for this study, the creep strain rate is independent of the prior stress history. Using the data of Figure 65 the apparent stress exponent 'n' can be determined for the power law creep equation, presented in section 2.5.1, but simplified to $\dot{\epsilon} = A\sigma^n$ since temperature is constant at 760°C. Both microstructures exhibit an increasing apparent stress exponent with increasing stress similar to other results[50]. Furthermore, consistent with the results of Figure 62, the furnace cooled material displays a higher strain rate at all stress levels tested.

6.2.2 Decremental Tests

In contrast to the incremental tests, the decremental tests involve periodic removal of a predetermined amount of the load and therefore a corresponding stress decrease, with the specific test procedure described in section 4.5. As discussed in section 2.5.3.1, the back stress present in a specific microstructure may be determined with the decremental test technique. To compare the effect of lamellar interface spacing on the back stress the two microstructures of Figures 50e and f were tested. A portion of the strain versus time curve is illustrated in Figure 66 showing the typical strain response when the stress is decreased for the furnace cooled sample of Figure 50f. Similar plots were obtained for further decrements but with longer incubation periods as the test proceeded, as in the schematic of Figure 34a. From this plot the incubation period was determined as the portion of the plot with a zero rate of creep strain as indicated on the Figure 66. As mentioned in section 2.5.3.1, to determine the back stress the cumulative incubation period is plotted versus the stress remaining on the specimen. A plot of this type is provided in Figure 67a for the fully lamellar microstructures. At the early stages of the test when the stress is still high, there are very short incubation periods observed following the first few load reductions. Because of the very slow strain rates it was often difficult to determine the time at which forward creep begins following a reduction. When the stress level is continuously reduced, at low stress the duration of the incubation periods of no straining increases. Replotting the results of Figure 67a as stress remaining versus incubation period following each stress level more clearly illustrates the different response in terms of incubation period following the stress drops. The division between these two different responses for the AC and FC microstructures occurs at ≈ 190 MPa and ≈ 130 MPa, respectively.

6.3 Deformed Microstructures

As was discussed before the air cooled specimen failed after $\approx 6.5\%$ strain, which is consistent with the brittle type of intergranular cracking observed, Figure 56b and 68a. This is compared to the furnace cooled microstructure which reached $\approx 18\%$ strain which is consistent with the highly deformed lamellar grains observed even at remote locations to the fracture surface. As well, for the furnace cooled structure several spherical voids were visible, in contrast to the brittle cracks at lamellar grain boundaries in the air cooled microstructure. In the furnace cooled structure there were also observed several shear bands within lamellar grains oriented with lamellae parallel to the load axis, Figure 68b. A shear band is formed when lamellae shear past each other at 45 degrees to the stress axis on the plane of maximum shear stress. Within the shear band spherodization of the α_2 plates is noted in Figure 68c. Spherodization involves breakup of the lamellae into spherical type particles.

The dislocation substructures observed after creep fracture of the air cooled specimen are illustrated in Figure 69. As mentioned in section 4.4.2, foils were tilted in a goniometer to determine the angle of lamellar grains to the load axis. A feature observed in the several air cooled foils was several bowed dislocations between lamellae, Figure 69a. The bowing of dislocations was observed within soft oriented grains, with the density of dislocations much lower in hard oriented grains and several γ lamellar exhibiting almost no change in dislocation density from the undeformed state, Figure 69b. For dislocations observed in hard mode grains less bowing was observed compared to soft mode grains, as well less deformation twinning was observed compared to the furnace cooled condition regardless of lamellar orientation.

To examine deformed substructures independent of the total applied strain a furnace cooled specimen was tested and the test stopped at 6.5% strain, the fracture strain of the air cooled specimen. The test was stopped with the load maintained as the

temperature was lowered to room temperature, to ensure the deformation structure did not recover. Foils of this specimen were examined and similar dislocation bowing segments were observed as with the air cooled specimen of Figure 69a with the bowed dislocations also occurring in soft mode grains. Generally, deformation structures consisted of soft mode grains with bowed and tangled dislocation arrangements, Figure 70a, while hard mode grains had less dense dislocation structures, Figure 70b and deformation twins occurring in widely spaced lamellae, Figure 70c. The furnace cooled specimen tested to failure shows a considerably higher dislocation density consistent with the increased strain ($\approx 18\%$), Figure 71.

6.4 Discussion of Creep for Microstructures with Variation in Lamellar Interface Spacing

The results of Figure 62, for the monotonic tests of the air and furnace cooled specimens illustrate that microstructure affects the creep properties of the air and furnace cooled microstructures, with the key differences being the lamellar interface spacing and the grain boundary morphology.

Considering first the results of the stress increment tests, we see that for the fully lamellar structures, the stress exponent increases with the increasing stress from ≈ 1 at low stresses to ≈ 10 at high stresses, Figure 65. Typically, this type of result is interpreted to indicate that the mechanism controlling the creep strain rate is dependent on stress. From Figure 65 at 240 MPa the stress exponents are ≈ 8 and ≈ 9 for the air and furnace cooled microstructures respectively. These stress exponents are in the range predicted for dislocation controlled creep[66]. The increased dislocation density observed from creep deformed material, Figures 69, 70, and 71, provides microstructural evidence that dislocation mechanisms influence the minimum creep strain rate at 240 MPa. The reduced lamellar interface spacing of the air cooled material, Figure 61, Table 2, may be

the microstructural parameter controlling dislocation creep, with evidence provided from the bowing of dislocations between adjacent lamellae, Figure 69a. Reducing the lamellar spacing would increase the stress required for glide of the bowed dislocations. This supports the idea, reviewed in section 2.4.3.3, of an Orowan stress explaining the increased creep resistance of the air cooled microstructure.

The stress decrement test results of Figure 67, indicate a boundary between the high stress region, which may be glide controlled, and the low stress region, which may be recovery controlled. This was stated to be at applied stresses (σ_a) of ≈ 190 MPa and ≈ 130 MPa, for the air and furnace cooled microstructures respectively. These values can be compared to the shear stress required for Orowan bowing of dislocations between lamellar interfaces as estimated according to [67]: $\tau_o = \frac{Gb}{L}$, where G is the shear modulus, b is the Burgers vector and L the γ lamellae width. Using typical values of the γ lamellae width of 250 and 600 nm for the air and furnace cooled conditions respectively yields an Orowan stress of 96 and 42 MPa. Assuming $\tau = \frac{\sigma_a}{2}$ gives similar measured Orowan bowing stresses of 95 and 65 MPa for the air and furnace cooled conditions respectively. Using this reasoning the σ_a values of 190 and 130 MPa can be interpreted as the internal stress required for dislocation glide.

The large strains in the furnace cooled specimen requires the generation of dislocations, which is illustrated in the deformed and undeformed samples, Figures 61, and 69. *In situ* TEM observations [68] have indicated that dislocation generation can occur at semi-coherent α_2/γ interfaces of near γ -TiAl alloys during deformation and that the ends of dislocation segments are associated with the same interface forming a loop. A similar phenomenon is postulated to occur in this study, as seen by Figures 69 and 70. These type of dislocations may grow outward until they intersect an adjacent interface of

a neighbouring lamellae, therefore forming a dislocation segment between interfaces as in Figure 69 and 70a.

As discussed in section 2.4.3.3 the characterization of dislocation behaviour with respect to the orientation of lamellar grains to the stress axis is of interest and may help explain deformation behaviour associated with near- γ TiAl lamellar alloys. Due to the anisotropy of the lamellar grains, the deformation characteristics vary significantly between grains, with soft oriented grains having a lower flow stress and therefore tending to deform first under the applied load. For soft grains the maximum shear stress direction is approximately parallel to the lamellar interfaces and there is therefore dislocation motion due to this stress. It was noted in 2.4.3.3 that for lamellar orientations between 30-50° strength of lamellar grains is low. However, the lamellar interfaces pin dislocation segments and constrain dislocation motion leading to the bowing of dislocations between lamellar interfaces observed in soft grains, Figure 69a and 70a, similar to Orowan bowing. The deformation of soft grains by dislocation motion is limited due to the resistance to deformation of the adjacent hard grains. The resulting strain discontinuity between hard and soft grains may be a reason for the low ductility associated with fully lamellar structures[69]. The hard grains therefore prevent further soft grain deformation until hard grains can deform. For continuing strain, hard grains deform so that the grain boundary incompatibility is reduced and soft grain deformation can occur by dislocation motion. The deformation of hard grains requires shear deformation across lamellar interfaces resulting in higher strength[69]. This supports the results that hard grains have greater resistance to deformation, which is consistent with the lower dislocation densities observed in Figures 69b and 70b. For the air cooled specimen, there is stable deformation of hard grains which allow a significant strain to be absorbed by the soft grains. This is supported by the nearly steady state creep rate observed in Figures 51 and 53. For the furnace cooled condition, the wider lamellae reduce the hard grain strength allowing deformation to occur more easily resulting in the faster strain rate observed for this microstructure.

Grain boundaries influence the strain discontinuity due to soft and hard grains deforming at different rates. For the air cooled condition the planar grain boundaries, Figure 50e, provide minimal resistance to intergranular cracking which results in rapid failure with minimal tertiary creep, Figure 51. In contrast the furnace cooled structure has interlocked lamellae along grain boundaries, Figure 50f, which provide resistance to intergranular crack propagation, but hard grains are more easily deformed due to the wider lamellar spacing. This leads to the formation of shear bands across hard grains, as shown in Figure 68. Similar shear band formation has been noted in other γ -TiAl alloys with well interlocked grain boundaries[70]. The resulting spheroidization of the lamellar structure leads to strain concentration along shear bands allowing easier deformation of the hard grains, allowing greater tertiary creep strain before final fracture. For the furnace cooled condition during tertiary creep, strain is accommodated by a combination of soft grain deformation by dislocation motion and by shear band formation in hard grains. As seen in Figure 68, there is void formation along shear bands, which coupled with the void formation at grain boundaries leads to reduction in area and final fracture.

CHAPTER 7

7. General Discussion

One of the primary objectives of this thesis work was to obtain a series of microstructures with a range of volume fractions of γ grains. From creep tests performed on these microstructures an effect on the creep behaviour due to the microstructure was found. γ grains surrounding larger lamellar grains cause grain boundary sliding (GBS) between γ grains. Dynamic recrystallization within deformed γ grains serves to increase the GBS and increase the creep rate. As the volume fraction of γ grains is increased the extent of GBS and dynamic recrystallization increase and the resistance to creep decreases. Several observations support this conclusion. Firstly, previous research has shown that dynamic recrystallization occurs in microstructures similar to those tested in this work[20,43,44]. Secondly, TEM results show evidence of dislocation free γ grains which may be newly formed dynamically recrystallized grains. The deformed γ grains surrounding the relatively intact lamellar grains provides support to the idea of GBS decreasing the creep resistance of nearly lamellar and duplex microstructures with high volume fractions (15-85%) of γ grains. In effect the γ grains are able to slide past the lamellar grains due to their smaller size and greater overall grain boundary area.

The nearly lamellar microstructure with a low volume fraction of γ grains (7-10%) may be the best microstructure for a combination of optimum mechanical properties. It may possess a balance of sufficient creep resistance and improved ductility compared to the fully lamellar structure.

The effect of lamellar spacing on creep behaviour was determined through a series of creep tests. Creep properties for the furnace cooled microstructure with wider spaced lamellae are inferior compared to the air cooled sample. From an analysis of deformed

structures, bowing of dislocations was apparent. For the air cooled structure, the reduced lamellar spacing leads to a higher Orowan shear stress for bowing of these dislocations. The value approximated for the Orowan bowing stress and that estimated from decremental creep data were found to be in close agreement for both air and furnace cooled microstructures(96 and 42 MPa and 95 and 65MPa respectively). Therefore, the resistance to dislocation glide offered by lamellar interfaces may be a factor leading to improved creep.

The deformed lamellar grains were also characterized according to the lamellar orientation to the stress axis. The soft grains have a higher dislocation density than hard grains, suggested to be due to the lower flow stress for the soft grains. A grain boundary incompatibility may also exist between adjacent hard and soft grains. The hard grains which are more resistant to deformation deform at a slower rate compared to the soft grains, leading to the incompatibility. This incompatibility may be relieved by the formation of shear bands in hard grains which would then allow further soft grain deformation to occur in the furnace cooled structure.

Fractured specimens displayed a brittle type failure for the air cooled specimen, while a more ductile type of failure was seen in the furnace cooled structure. It was suggested that the low ductility may be the result of the strain incompatibility at the grain boundary previously discussed, or it may be due to the morphology of the grain boundary itself. The planar grain boundaries of the air cooled structure provide minimal resistance to intergranular crack propagation while the more interlocked grain boundaries of the furnace cooled structure lead to a more tortuous crack path. This grain boundary effect combined with the wider spaced lamellae lead to a higher overall creep fracture strain and a higher creep strain rate.

CHAPTER 8

8. Summary & Conclusions

1. A review of current literature pertaining to near γ -TiAl is presented, with the emphasis on microstructural development, and the creep behavior of lamellar microstructures.
2. The γ grain volume fraction can be varied by controlled heat treatments near the α transus temperature. The γ grain volume fraction is dependent on both heat treatment time and temperature.
3. A definite trend exists indicating decreasing creep resistance as the volume fraction of γ grains increased. This results from dynamic recrystallization within the γ grains leading to increased grain boundary sliding.
4. Lamellar interface spacing is controlled by the cooling rate following heat treatment in the α phase region. Slow cooling results in wider interface spacing.
5. Creep tests on the microstructures with different lamellar interface spacing indicate that a wider spacing results in a creep rate 5 times as fast as the narrower spacing. As well, the creep life for the wider spacing is half that of the narrower air cooled structure.
6. Narrower lamellar interface spacing reduces the creep strain rate due to interaction between dislocations and interface boundaries that inhibit dislocation motion.
7. Interlocking lamellae along grain boundaries improve resistance to intergranular cracking, allowing greater total creep strain and potentially greater creep life.
8. Several features of the creep deformed microstructures indicate that the creep response of individual lamellar grains is dependent on the lamellar orientation to the stress axis.

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Table 1 - Comparison of Gamma TiAl with Titanium Alloys and Superalloys

Property	Ti-Alloys	γ -TiAl	Superalloys
Density (g/cm ³)	4.54	4.04	8.3
Stiffness (GPa)	110	176	207
Max T - Creep (°C)	540	900	1090
Max T - Oxidation (°C)	590	815	1090
Ductility - Room T (%)	15	1-3	3-10
Ductility - Oper. T (%)	15	5-12	10-20

Table 2 - Microstructural Parameters and Creep Results

Heat Treatment *	Gamma Grain Volume Fraction (%)	Gamma Grain Size (μm)	Lamellar Grain Size (μm)	Average Lamellar Spacing (nm)	Minimum Creep Rate (h ⁻¹)	Time to Fracture (h)	Fracture Strain (%)
(DP) 1350°C/2h/A.C	80-85	<35	60	-	1.7x10 ⁻³	68.5	36.4
(NL) 1365°C/1h/A.C	20-25	<30	94	-	7.8x10 ⁻⁴	145.5	23.4
(NL) 1370°C/1h/A.C	15-20	<25	170	-	3.6x10 ⁻⁴	255	20.5
(NL) 1375°C/1h/A.C	7-10	<20	234	-	1.2x10 ⁻⁴	455	11.7
(FL) 1380°C/1h/A.C	<1	<5	290	120	6.0x10 ⁻⁵	608	6.5
(FL) 1400°C/1/2h/F.	<1	<10	325	450	2.7x10 ⁻⁴	279	18.4

*additional 950°C/48h heat treatment applied to all specimens

a

b

Figure 1 5th stage turbine blades run in CF6 engine a) before testing b) after approximately 1000 engine cycles.[3]

Figure 2 Exhaust valves developed by Ford motor company showing valves produced by investment casting, permanent mold casting and a finished valve.[7]

γ -TiAl: Crystal structure

Figure 3 $L1_0$ crystal structure of γ -TiAl and schematic illustrating dislocation dissociation.[8]

Ti₃Al: Crystal structure

Figure 4 D0₁₉ crystal structure of α_2 -Ti₃Al, and schematic of crystallographic planes.[8]

Figure 5

a) Phase diagram for Ti-Al system.

b) Expanded view of central portion of Ti-Al phase diagram. Symbols T_1 , T_2 , T_3 , and T_4 , correspond to heat treatment temperatures with resulting microstructures given in Figures 7a to d.

Figure 6 Effect of chromium on α transus, showing a shift in α transus temperature with chromium addition.[14]

Figure 7 Resulting microstructures from heat treatments described in Figure 5b
 a) Duplex microstructure
 b) Near γ equiaxed microstructure
 c) Nearly lamellar microstructure
 d) Fully lamellar microstructure.[47]

2

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PM-1 3 1/2" x 4" PHOTOGRAPHIC MICROCOPY TARGET
NBS 1010a ANSI/ISO #2 EQUIVALENT

PRECISIONSM RESOLUTION TARGETS

Figure 8 a) Nearly lamellar microstructure with γ grains predominantly at g.b.'s [19]
 b) Nearly lamellar microstructure with γ grains at grain interior. [19]

Figure 9 Relationship between the interface spacing and the cooling rate for lamellar structures formed in Ti-48Al [17]

Figure 10 a) Air and b) furnace cooled microstructures.[20]

Figure 11 a) Grain boundary morphology of Ti-48Al after 1375°C heat treatment for 100 hours b) 25 hours. [19]

Figure 12 Room temperature tensile properties of a two-phase γ alloy in various heat-treatment conditions.[15]

Figure 13 Variations of tensile properties with test temperature for various microstructural conditions.[15]

Figure 14 The effective local fracture strain in the plastic zone for a near γ -TiAl alloy at room temperature [25]

Figure 15 Shear ligament in fully lamellar microstructure.[28]

Figure 16 a) Fatigue behaviour at high temperatures in air comparing fully lamellar and duplex conditions.[25]
b) Fatigue crack growth rates for duplex and fully lamellar conditions.[25]

Figure 17 Creep results for single phase γ constant stress tests for Ti-53.4Al at 827°C a) 251 MPa b) 126 MPa.[31]

Figure 18 Creep results for single phase γ constant stress tests for Ti-51.5Al at 827°C a) 251 MPa b) 100 MPa.[32]

Figure 19 Creep results for single phase γ constant stress tests for Ti-50Al at 827°C and 251 MPa.[33]

Figure 20 Minimum creep rate as a function of stress in single-phase TiAl.[34]

Figure 21 Temperature compensated creep results versus applied stress minimum threshold stress for Ti-50 alloy.[38]

Figure 22 Comparison of creep properties for single phase γ , duplex, and fully lamellar microstructures tested at 815°C and 87.5 MPa.[40]

Figure 23 Creep curves showing effect of grain size on tertiary creep for Ti-48Al+ W.[16]

Figure 24 a) 20min/1400°C F.C.[42]
b) 20min/1400°C A.C.[42]
c) 2h/1400°C F.C. [20]

Figure 25 Creep curves of PM-Ti48Al3Cr heat treated to obtain a range of lamellar volume fractions.[45]

Figure 26 Creep deformation of fully lamellar microstructure, showing dislocations pinned at interface boundaries. [40]

a

b

Figure 27 Properties of lamellar single crystals as a function of lamellar orientation to the applied load.
a) tensile elongation as a function of lamellar orientation to applied load
b) yield strength as a function of lamellar orientation to applied load [51]

Figure 28 Steady-state creep rate as a function of applied stress at 760°C [50]

Figure 29 a) Stress reduction tests in the high stress region.[50]
b) Stress reduction tests in the low stress region.[50]

Figure 30 Schematic representation of a creep curve.[59]

Figure 31 Schematic representation of $\ln(\dot{\epsilon}_s)$ versus $(1/T)$ where the gradient represents $(-Q_c/R)$, where Q_c is the activation energy for creep and R is the universal gas constant.[36]

Figure 32 Schematic representation of $\log(\dot{\epsilon}_s)$ versus $\log(\sigma)$ where the gradient is the stress exponent 'n' used in the power-law creep equation.[36]

Figure 33 Dislocations gliding and climbing over an obstacle. [36]

a

b

Figure 34 a) Schematic plot of incubation periods associated with a stress decrement test.[58]
b) Schematic of cumulative incubation time with cumulative stress decrements.[58]

Figure 35 Schematic of a constant load creep frame.[36]

Figure 36 Extensometer assembly used to hold a creep specimen.[36]

Figure 37 Schematic representation of the TGA process.[60]

Figure 38 **Vacuum system for hermetically sealing titanium tubes containing powder.**

Figure 39 **Glove box used for preparation of powder filled tubes.**

Figure 40 Tube filled with powder being hydraulically crimped in preparation for sealing.

Figure 41 Schematic representation of a hot isostatic press.

Figure 42 HIP cycle for consolidation of Ti-48.1%Al powder.

Figure 43

- a) TiAl powder
- b) titanium tubing crimped and sealed at one end
- c) filled and crimped titanium tubing ready for HIP'ing.
- d) HIP'ed specimen (note slightly narrower diameter after consolidation)
- e) rods of TiAl after machining
- f) vacuum encapsulated tube prepared for heat treatment
- g) machined creep specimen.

Figure 44 Creep testing frame.

Figure 45 Calibration curve used to determine mm/volt for use in calculating specimen strain.

Figure 46 Comparison of creep curves for the same microstructure.[62]

Figure 47 Central portion of phase diagram for Ti-Al.

Figure 48

As HIP'ed microstructure of Ti-48Al. HIP'ed at 200MPa, 1250°C for 2 hours.

Figure 49

As HIP'ed microstructure illustrating prior dendritic structure.

Figure 50

Microstructures prepared for creep testing.

- (a) Duplex (DP) 1350°C/2h/A.C., volume % γ = 80-85%
- (b) Nearly Lamellar (NL) 1365°C/1h/A.C., volume % γ = 20-25%
- (c) Nearly Lamellar (NL) 1370°C/1h/A.C., volume % γ = 15-20%
- (d) Nearly Lamellar (NL) 1375°C/1h/A.C., volume % γ = 7-10%
- (e) Fully Lamellar (FL) 1380°C/1h/A.C., volume % γ = < 1%
- (f) Fully Lamellar (FL) 1400°C/1/2h/F.C., volume % γ = < 1%.

Figure 51 Results of creep tests at 760°C and 240 MPa for microstructures of Figures 50a to e in percent engineering strain versus time.

a)

b)

Figure 52 (a) Time to fracture versus the volume fraction of γ grains (b) Minimum creep strain rate versus volume fraction γ grains.

Figure 53 Strain rate versus strain for FL and NL microstructures of Figure 50 d and e.

Figure 54 1365°C/1h/A.C. heat treated condition (a) before deformation (b) after creep failure

Figure 55 Creep failures indicating cracks formed at lamellar γ grain locations for a) 1365°C/1h/A.C. heat treated condition (b) 1370°C/1h/A.C. heat treated condition.

a

b

Figure 56 Creep failures for indicating brittle intergranular cracks formed at lamellar grain boundaries a)1375°C/1h/A.C. heat treated condition (b)1380°C/1h/A.C. heat treated condition.

a)

b)

c)

Figure 57 TEM images of a) undeformed DP microstructure with lamellar and γ grains present $\times 10500$ b) dislocations observed within a lamellar grain $\times 34000$ c) dislocations observed in a γ grain $\times 34000$. (b) and c) after creep deformation)

a)

b)

Figure 58

a) b) Dislocation free γ grains observed in DP microstructure after creep failure

Figure 59 Creep deformed NL (15-20% γ grains) a) dislocations observed within a lamellar grain x34000 b) dislocations observed in a γ grain x34000.

Figure 60 Dislocation free γ grain observed in NL microstructure of Figure 50c x23000. (after creep failure)

Figure 61 TEM microstructures of a) $1380^{\circ}\text{C}/1\text{h}/\text{A.C.}$ b) $1400^{\circ}\text{C}/1/2\text{h}/\text{F.C.}$ Both microstructures stabilized at $950^{\circ}\text{C}/48\text{h}$.

Figure 62 Results of creep tests at 760°C and 240 MPa for air and furnace cooled specimens (Figures 50e and f) respectively in percent engineering strain versus time. Included is the furnace cooled test interrupted at 6.5 % strain.

Figure 63 Strain rate versus strain for AC and FC microstructures of Figure 50c and f, illustrating lack of steady state strain rate for FC microstructure.

Figure 64 Stress increment test result for $1400^{\circ}\text{C}/1/2\text{h}/\text{F.C.}$ specimen.

Figure 65 Comparison of stress increment test results.

Figure 66 Stress decrement result for the air cooled microstructure, showing the incubation period after a stress decrease.

Figure 67 a) Stress remaining versus cumulative incubation period for the fully lamellar microstructures of Figures 50e and f.

Figure 67 b) Stress remaining versus non cumulative incubation period for the fully lamellar microstructures of Figure 50e and f.

Figure 68

Microstructure of 1380°C/1h/A.C. heat treated condition after creep failure showing brittle cracking b) Microstructure of 1400°C/1/2h/F.C. material after creep failure exhibiting void formation and shear bands. c) Spherodization of the α_2 plates of the 1400°C/1/2h/F.C microstructure.

Figure 69 Deformed microstructure of 1380°C/1h/A.C. condition after creep failure. a) soft oriented grain and b) hard oriented grain. Arrows indicate dislocation bowing from lamellar interfaces.

Figure 70 Deformed microstructure of 1400°C/1/2h/F.C. condition after 6.5% strain. a) soft oriented grain and b) hard oriented grain and c) hard oriented grain exhibiting deformation twinning. Arrows indicate dislocations emanating from lamellar interfaces.

Figure 71 Deformed microstructure of 1400°C/1/2h/F.C. condition after creep failure.

END

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