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A Heat Treatment Procedure to Produce Fine-Grained Lamellar Microstructures in a P/M Titanium Aluminide Alloy

by Peter Au, M.A.Sc.

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

Doctor of Philosophy

Department of Mechanical and Aerospace Engineering

Ottawa-Carleton Institute
for Mechanical and Aerospace Engineering

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submitted by
Peter Au, M.A.Sc.

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ABSTRACT

A process for fabricating advanced aerospace titanium aluminide alloys starting from metal powders (the hot isostatically consolidated P/M process) is presented in this thesis. This process does not suffer the difficulties of chemical inhomogeneities and coarse grain structure of castings. In addition heat treatments which take advantage of the refined structure of HIP processed materials are developed to achieve microstructure control and subsequent mechanical property control. It is shown that a better “property balance” is possible after the heat treatment of HIP consolidated materials than it is with alternative processing.

It is well understood that the standard microstructures (near- γ , duplex, nearly lamellar, and fully lamellar) do not have the balanced mechanical properties (tensile, yield, creep and fatigue strength, ductility and fracture toughness) necessary for optimal performance in aero engine and automotive applications. In this work a fine-grained fully lamellar (FGFL) microstructure is developed for property control and in particular for achieving a much improved property balance. A heat treatment procedure for this purpose which consists of cyclic processing in the α transus temperature region to achieve an FGFL structure with grain sizes in the range of 50 μm to 150 μm is presented. Compared with conventional duplex structured materials, the minimum creep rate is an order of magnitude lower with only a 10% loss in tensile yield strength. Moreover, a three-fold increase in tensile elongation is possible by converting to an FGFL structure with only a 30 % loss in minimum creep rate. These are attractive trade-offs when considering the use of these alloys for aerospace purposes.

A thorough literature review of the mechanisms of formation of standard microstructures and their deformation under mechanical loading is contained in the thesis. In addition, conventional techniques to produce FGFL microstructures in wrought and cast materials are discussed in detail. Beyond the review, the results of experiments are described for determining the α transus temperature, the phase transformation kinetics in this region and the effects of heat treatment time and cooling rate on microstructure. Based on this preliminary work, a heat treatment to achieve a FGFL microstructure with grain sizes in the range of 50 μm to 150 μm is proposed and confirmed. The room temperature and high temperature mechanical properties of these materials are compared with those of conventional duplex and fully lamellar structures. The results of this experimentation are discussed in terms of the fundamental mechanisms for controlling microstructure and mechanical properties in these materials. The potential for applying cyclic heat treatments to cast and wrought materials to improve the mechanical property balance in engineering practice is discussed.

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NOMENCLATURE

Symbol	Description
A	microstructure-dependent constant in power law creep equation
AC	air cool
DPLX	duplex microstructure
DPEQX	dual-phased equiaxed microstructure
FC	furnace cool
FGFL	fine-grained fully lamellar
FL	fully lamellar microstructure
HIP	hot isostatically pressed
K_{IC}	plane-strain fracture toughness, $\text{MPa}\sqrt{\text{m}}$
L_{gb}	lamellar grain boundaries
L_{gs}	average lamellar grain size, μm
L_s	start of lamellae formation
$T_{0.1.2...}$	heat treatment temperature, $^{\circ}\text{C}$
Q	activation energy, kJ/mol
T	absolute temperature in power law creep equation, K
R	Universal gas constant, cal/mol/K
T_{α}	alpha transus temperature, $^{\circ}\text{C}$
$V_{\gamma g}$	volume fraction of gamma grains, %
WQ	water quench
YS	0.2% yield stress, MPa
a,c	lattice parameters, nm
d	grain size in Hall-Petch relation, m
k	constant in parabolic grain growth equation
k_y	constant in Hall-Petch relation
n	exponent in parabolic grain growth equation, or in power law creep equation
t	heat treatment time in parabolic grain growth equation, minutes
α	disordered hexagonal close-packed alpha phase
α_2	ordered hexagonal close-packed alpha phase
β	disordered body-centred cubic beta phase
β_2	ordered body-centred cubic beta phase
ε	engineering strain, %
$\dot{\varepsilon}_{\min}$	minimum engineering strain rate, h^{-1}
γ	ordered face-centred tetragonal gamma phase
γ_{gs}	average gamma grain size, μm
γ_s	start of gamma phase transformation
γ_m	massively transformed gamma phase
γ_{ms}	start of massive transformation of gamma phase
θ	orientation of lamellae with respect to the loading direction, in degree
σ	stress, MPa

CHAPTER 1

INTRODUCTION

Titanium aluminide intermetallic alloys (TiAl) have a number of attractive properties, including low density, excellent retention of strength and stiffness at high temperatures, and good creep, oxidation and burn resistance. Because of this, these materials are potential replacements for titanium alloys and superalloys in automotive and aero engine structural applications. Some of the potential uses of TiAl in aero engines are shown diagrammatically in Figure 1 [1].

There are two classes of TiAl alloys of interest: the alpha-2 alloys (α_2 -Ti₃Al) which contain 22 to 25% aluminum (compositions are expressed in atomic per cent unless otherwise stated), and the near-gamma alloys (γ -TiAl) which contain 46 to 52% aluminum. Between the two classes of titanium aluminides, the γ -TiAl alloys have better high temperature properties and therefore better potential for improving engine performance and efficiency. This thesis will focus on the γ -TiAl alloys.

The properties of γ and α_2 alloys are compared with those of other superalloys and titanium alloys listed in Table 1 [2-4]. It can be seen that the intermetallics have densities approximately half of those of the superalloys, and exhibit better high temperature creep and oxidation resistance than their titanium alloy counterparts, but their room temperature ductility and fracture resistance are relatively poor. In the past several years, the potential of γ -TiAl alloys has been demonstrated by successful engine tests that involved automotive turbo-charger rotors and exhaust valves [5,6], as well as aero engine low pressure turbine

blades [7]. However, there are still many obstacles preventing the implementation of γ -TiAl materials on a production scale, one of which is the lack of balance in mechanical properties including tensile ductility, fracture toughness, and creep properties. As a result, considerable effort has been devoted to overcome these shortcomings by compositional modification and microstructural control.

The mechanical properties of γ -TiAl alloys depend on their microstructures. A variety of microstructures can be produced by different processing methods, heat treatment schemes, thermomechanical treatments, and alloy composition modifications. Typical processing routes for γ -TiAl alloys are shown in Figure 2. Among these processing methods, investment casting and wrought processing of ingot metallurgy materials have received the most attention because of their potential for low-cost high-volume production, whereas powder metallurgy processing has received much less attention partly due to the relatively high cost of powder materials. However, the microstructure of as-cast materials is inherently non-uniform, coarse-grained and has an especially poor balance of mechanical properties. Therefore, the alloys typically have to be processed further with combinations of thermomechanical and heat treatments to reduce compositional inhomogeneities and to refine the microstructure. On the other hand, powder metallurgy processed materials have a much more homogeneous and refined starting microstructure, which is more amenable to hot work and microstructural design to achieve desired mechanical properties [8, 9].

In general, the microstructures of γ -TiAl alloys can be divided into four ‘standard’ types, as shown in Figure 3 :

1. a “near- γ ” equiaxed microstructure that consists predominantly of γ grains with a small volume fraction of α_2 phase particles. Figure 3(a). This structure is associated with alloys containing approximately 50 % aluminum and above.
2. a uniform fine-grained “duplex” microstructure that consists of single-phase equiaxed γ and lamellar grains with a typical grain size of 30 μm , Figure 3(b). This microstructure can be produced generally by hot working cast materials, or by powder metallurgy processing.
3. a “nearly lamellar” microstructure that consists mostly of coarse lamellar grains with a small volume fraction of equiaxed γ grains distributed predominantly at the grain boundaries. Figure 3(c). This microstructure has been produced by hot working cast materials.
4. a coarse-grained “fully lamellar” microstructure with a typical grain size larger than 500 μm , Figure 3(d). This structure is commonly found in cast materials.

In these last three classes of microstructure, the lamellar grains consist of alternating platelets of the $\alpha_2 + \gamma$ phase constituents which are formed from the melt by a sequence of peritectic and eutectoid transformations as indicated by line “C” in Figure 4(b). These reactions are described in more detail in 2.2.4.

A schematic comparison of the mechanical properties of standard microstructures is presented in Figure 5. The “near- γ ” and “duplex” microstructures have good room temperature tensile properties but poor fracture toughness and creep resistance, whereas the reverse is true for the coarse-grained “fully lamellar” microstructure. The nearly lamellar

microstructure offers a somewhat better combination of properties, but its creep and fracture resistance are inadequate for gas turbine engine applications. Very recently, it has been shown that a uniform “fine-grained fully lamellar” (FGFL) microstructure improves the mechanical property balance in γ -TiAl alloys, although the formation mechanisms and the deformation behaviour of this microstructure are not fully understood [9-18]. Because of the nature of the phase transformations in near- γ alloys, it is difficult to produce FGFL structure by conventional heat treatments. The FGFL microstructures reported so far have been produced by combinations of elaborate thermomechanical processing [9-11], the addition of grain refinement agents such as boron or TiB_2 particles [12-15], and innovative heat treatments [16-18]. The possibility of producing a FGFL microstructure in powder metallurgy (P/M) materials has only been explored recently [9]. Unlike ingot metallurgy and investment cast materials, P/M materials have a fine and homogeneous starting microstructure, which can be an important advantage to achieve a FGFL microstructure. Also, the technique and knowledge developed using P/M alloys might possibly be extended to cast and wrought materials. Therefore, the objective of this thesis is to develop a heat treatment scheme to produce fine-grained fully lamellar microstructure using a near- γ Ti-47.5Al-3Cr P/M material. The formation mechanisms, tensile and creep properties, and deformation mechanisms of the FGFL microstructure are investigated and compared with those of the duplex and fully lamellar microstructures.

The layout of the thesis is as follows: Chapter 2 provides a literature review; Chapter 3 outlines the experimental procedure; Chapter 4 describes the experimental results; detailed and general discussions of the results are presented in Chapter 5 and 6, respectively; summary and conclusions are outlined in Chapter 7.

CHAPTER 2

LITERATURE REVIEW

Gamma titanium aluminides have a long range ordered crystal structure that persists at high temperatures. This characteristic enables them to retain their strength and stiffness at elevated temperatures. However, it also contributes to their relatively poor ductility and fracture resistance at room temperature. Therefore considerable effort has been devoted to balancing these properties by controlling the microstructure via compositional adjustments, thermomechanical processing and heat treatment. This chapter presents an overview on this subject. The phase diagram of binary near-gamma alloys and the basic structures of the major phases are presented, and the classes of alloys of current interest are described. The relationships between processing, heat treatment and the microstructures, mechanical properties and associated deformation mechanisms are also highlighted.

2.1 Phase Diagram And Crystal Structures

Near- γ TiAl alloys with potential engineering applications are multicomponent alloys that typically contain 46 to 49% aluminum and up to 10% of other elements including B, Cr, Mn, Mo, Nb, Si, Ta, V and W. The objectives of these elemental additions are to improve the strength and ductility, workability, oxidation and creep resistance of these alloys [10,19-22]. Details on the effects of various elements will be presented in a later section. Near- γ TiAl alloys can be divided generally into two groups: single-phase γ alloys containing 50-55 % aluminum, and two-phase $\gamma+\alpha_2$ alloys containing 45-49 %

aluminum. Phase diagrams for multicomponent alloys are generally not available. In fact, the central portion of the Ti-Al binary phase diagram has only been developed recently and more work is required to improve its accuracy [23-25]. However, for an understanding of the basic phase transformation processes that control the microstructure of these alloys, the binary Ti-Al equilibrium phase diagram is adequate.

The binary Ti-Al phase diagram [26] and its enlarged central portion are shown schematically in Figure 4. Several regions in the phase diagram are important for heat treatments and microstructural control:

- the high temperature ($\beta+\alpha$) two-phase region,
- the high temperature α single-phase field,
- the ($\alpha+\gamma$) two-phase field bounded by the α transus line ' T_α ' and the γ solvus line ' γ_S ',
- and the $\alpha_2+\gamma$ two-phase field.

Four solid phases are of interest: the high temperature disordered beta phase ' β -Ti' and alpha phase ' α -Ti', which transform to ordered phases at lower temperatures. The ordered alpha-2 phase ' α_2 -Ti₃Al' and the ordered gamma phase ' γ -TiAl' are the major constituents in near- γ TiAl. Depending on the temperature, the composition of α_2 and γ can vary from 22 to 39% aluminum and from 49 to 66% aluminum, respectively. The α_2 phase has a DO₁₉ ordered hexagonal structure, whereas the γ phase has an L1₀ ordered face-centred tetragonal structure with a tetragonality c/a of 1.02 at the equiatomic composition. This ratio increases to 1.03 at higher aluminum content. The lattice structure, possible slip

planes and directions of the γ phase and the α_2 phase are shown in Figure 6 and Figure 7. respectively.

2.1.1 Effects of Ternary Element Additions

It has been reported that ternary additions of elements such as Cr, Mn, and V decrease the c/a ratio and unit cell volume of the γ phase by occupying the aluminum site. This leads to a weakening of the Ti-Al covalent bond and an increase in the propensity for twin deformation, resulting in improved room temperature ductility [27]. Ternary additions of these elements can also lower the α transus line ' T_α ' in the ternary phase diagram. For example, it has been observed that for binary TiAl containing 45-50% aluminum, the values of T_α are reduced by 12-17 °C for each at.% addition of Mn, Cr and V, Figure 8 [20].

It has been suggested that additions of ' β stabilising' elements such as W, Cr and Nb, expand the ($\beta+\alpha$) two-phase field in the phase diagram to lower temperatures and shift the ($\beta+\alpha$)/ α phase boundary toward the aluminum rich side. As a result, heat treating in this expanded field can refine the microstructure because the presence of disordered β phase inhibits the growth of α grains [11]. At lower temperatures, the disordered β phase transforms to ordered body centred cubic β_2 phase. The presence of ordered β_2 phase at γ grain boundaries can reduce grain growth during heat treatment, as well as promoting superplastic behaviour by spreading along the γ grain boundaries during isothermal forging [28-30].

2.2 Microstructures

The single-phase alloys, which contain 50-55 % aluminum, typically consist of equiaxed γ grains approximately 50 μm in diameter [25]. These alloys attracted attention initially because of their excellent resistance to oxidation and hydrogen absorption. However, they exhibit poor room temperature ductility (<1% elongation) and fracture toughness. Attempts to improve these properties by elemental additions of V, Nb and W and heat treatments have been unsuccessful [31]. As a result, interest in single-phase alloys is quite limited at the present time. In contrast, two-phase alloys that contain 45-49% aluminum have been the focus of attention because a variety of microstructures and therefore mechanical properties can be obtained by different processing routes and compositional modifications. The following discussion concentrates on the two-phase alloys.

The microstructures of two-phase alloys that have been investigated can be broadly divided into four standard types: near-gamma, fine-grained duplex, near fully lamellar, and coarse-grained lamellar. Beside the standard microstructures, a fine-grained lamellar microstructure has also been recently reported [9-18]. As it will become evident later, the fine-grained lamellar microstructure appears to have the best mechanical property balance. In the following sections, the characteristics and formation mechanisms of the aforementioned microstructures are presented, and the general attributes and shortcomings of some of their mechanical properties are briefly mentioned. More details on the mechanical properties and deformation mechanisms are presented in a later section.

2.2.1 Near-Gamma Microstructure

When an alloy with a composition “C” in Figure 4(b) is aged for a long duration of time at temperatures T_0 in the $(\alpha_2+\gamma)$ two-phase field, or at T_1 in the $(\alpha+\gamma)$ two-phase field, a non-uniform ‘near- γ ’ microstructure that consists predominantly of equiaxed γ grains with small α_2 grains distributed at the grain boundaries is obtained, Figure 3(a). Although this microstructure has good room temperature tensile properties, its fracture toughness and creep resistance are poor [32-34], it has therefore received little attention compared to the other microstructures.

2.2.2 Duplex Microstructure

Two types of duplex microstructures have been developed: a ‘cast-duplex’ microstructure produced in cast materials, and a fine-grained duplex microstructure produced in wrought materials and powder metallurgy processed materials.

A cast-duplex microstructure can be obtained by annealing the as-cast material in the $(\alpha+\gamma)$ two-phase field for at least 20 to 50 hours [15,17,27], during which γ grains are recrystallized predominantly at the lamellar grain boundaries. The resulting microstructure is somewhat refined and consists of a non-uniform mixture of equiaxed γ grains and lamellar grains, Figure 9. A room temperature ductility of 2.3% has been reported for a Ti-48Al-2Nb-2Cr alloy with a cast-duplex microstructure [15]. However, depending on the alloy composition, annealing alone cannot always refine the as-cast structure within a reasonable period of time. In most cases, thermomechanical treatment (TMT) such as isothermal forging is needed to break up the as-cast structure and facilitate dynamic

recrystallization. Before TMT, a homogenization treatment is usually carried out at temperatures either in the single-phase α field or in the $(\alpha+\gamma)$ two-phase field to reduce compositional segregation. After homogenization, the material is hot worked at temperatures in either the $(\alpha+\gamma)$ or the $(\alpha_2+\gamma)$ two-phase field [35-43], or as suggested very recently, in the $(\alpha+\beta)$ two-phase field [11], to break up the as-cast lamellar structure and dynamically recrystallize γ and α_2 grains. An example of a cast and forged microstructure is shown in Figure 10, which shows a banded mixture of γ grains, stringers of α_2 particles, and residual lamellar grains. The banded morphology can be eliminated by multidirectional hot working and annealing the material repeatedly. When a hot worked material with a uniformly recrystallized microstructure is heat treated in the $(\alpha+\gamma)$ field and air cooled to room temperature, a uniform fine-grained duplex microstructure that consists of a mixture of lamellar and equiaxed γ grains ranging from 10 to 40 μm in diameter can be obtained.

Compared with cast and wrought materials, powder metallurgy materials are considerably more suitable for producing a uniform fine-grained microstructure. This can be attributed to their inherently homogeneous and refined microstructure after consolidation [8,9,44-52]. A fine-grained duplex microstructure can be obtained simply by post-consolidation heat treatment in the $(\alpha+\gamma)$ field and air cooling to room temperature. An example of this is shown in Figure 11.

The grain size and volume fraction of the lamellar and γ grains in a duplex microstructure depend on the heat treatment temperature. Referring to Figure 4(b), heat treating at T_2 is expected to produce a uniform microstructure with approximately equal

volume fractions of lamellar and equiaxed γ grains because at this temperature, the initial quantities of α and γ phases are approximately equal, resulting in competitive growth of α and γ grains. Upon cooling, α_2 and γ lamellae are formed in the prior α grains via the following sequence [53]:

Heat treating at T_3 is expected to result in a lower volume fraction of equiaxed γ grains and a larger lamellar grain size because the α phase predominates at this temperature. In contrast, heat treating at T_1 is expected to produce a higher volume fraction of equiaxed γ grains and fewer lamellar grains. It has been suggested that a lamellar grain volume fraction of approximately 30% yields the best balance in tensile properties and creep resistance [19]. Compared to other standard microstructures, the duplex microstructure has better room temperature tensile strength and ductility (up to 4% elongation), but inferior creep resistance and fracture toughness (10-16 MPa $\sqrt{\text{m}}$).

2.2.3 Near Fully Lamellar Microstructure

When heat treatments are carried out in the $(\alpha+\gamma)$ two-phase field at temperatures slightly below the alpha transus temperature where α phase predominates, e.g. at T_3 in Figure 4(b), a near fully lamellar microstructure is produced [34,43]. This microstructure consists primarily of coarse lamellar grains that are $\geq 200 \mu\text{m}$ in diameter, and a small amount of equiaxed γ grains that are mostly located at the lamellar grain boundaries, giving rise to a “necklace” appearance, Figure 12. The lamellae in this microstructure are formed in the same manner as in the fully transformed microstructure, which is described in the

next section [53]. The near fully lamellar microstructure initially attracted some attention because its room temperature tensile strength is higher than that of the standard fully lamellar microstructure, and its ductility is marginally less than that of the duplex microstructure. This led to the expectation that a near fully lamellar microstructure would also provide a good mechanical property balance. However, it was later found that the fracture toughness and high temperature strength were inadequate for many applications [10,32,53], and that the volume fraction of the equiaxed γ grains in this microstructure is difficult to control using conventional heat treatments. Interest in this microstructure has therefore diminished.

2.2.4 Fully Lamellar Microstructure

When cast or wrought two-phase alloys having the composition 'C' as shown in Figure 4b are heat treated at temperatures above T_α in the α single-phase field (e.g. at T_4) and furnace cooled, a coarse-grained fully lamellar microstructure is produced, Figure 13(a). The grain size of this microstructure is typically $>500 \mu\text{m}$; a small amount of equiaxed γ grains is sometimes present at the grain boundaries. This type of microstructure has received a lot of attention because relative to other standard microstructures, it has superior fracture toughness and creep resistance, although its room temperature ductility is poor.

The lamellae in this microstructure are made up of alternating layers of α_2 and γ laths that are crystallographically aligned in such a way that the close-packed planes and directions in the γ phase and the α_2 phase are parallel, i.e. $\{111\}\gamma // (0001)\alpha_2$ and $\langle 110 \rangle \gamma //$

$\langle 1120 \rangle \alpha_2$ [54]. A TEM image of α_2 and γ plates in a lamellar grain is shown in Figure 13(b). It has been proposed that at moderate cooling rates (e.g. furnace cooling), the formation of the lamellae can follow three different routes [53]:

Type 1: $\alpha \rightarrow \alpha + \gamma_{\text{plates}} \rightarrow L(\alpha/\gamma) \rightarrow L(\alpha_2/\gamma)$

Type 2: $\alpha \rightarrow \alpha_2^s + \text{stacking faults} \rightarrow \alpha_2 + \gamma_{\text{precipitates}} \rightarrow \alpha_2 + \gamma_{\text{plates}} \rightarrow L(\alpha_2/\gamma)$

Type 3:

(a) $\gamma_{\text{matrix}} + \alpha_{2\text{particles}} \xrightarrow{\text{heating}} \gamma_{\text{matrix}} + \alpha_{\text{particles}} \rightarrow \gamma_{\text{matrix}} + \alpha_{\text{plates}} \rightarrow L(\gamma/\alpha) \xrightarrow{\text{cooling}} L(\gamma/\alpha_2)$

(b) $\gamma_{\text{matrix}} \xrightarrow{\text{heating}} \gamma_{\text{matrix}} + \alpha_{\text{precipitates}} \rightarrow \gamma_{\text{matrix}} + \alpha_{\text{plates}} \rightarrow L(\gamma/\alpha) \xrightarrow{\text{cooling}} L(\gamma/\alpha_2)$

The formation of the lamellae in alloys with 44 to 49% aluminum generally follows the first path. In this path, upon cooling from casting or during heat treatment in the α single-phase field, γ phase in the form of thin plates begins to precipitate out of the disordered α matrix, giving rise to the high temperature lamellar structure $L(\alpha/\gamma)$. This structure transforms to the low temperature lamellar structure, $L(\alpha_2/\gamma)$, by the ordering of α to α_2 . The lamellar spacing of this structure is typically less than 0.2 μm .

Alloys with an aluminum content in the range of 40 to 44 % tend to follow the second route. In this route, as the disordered α phase is cooled from the α single-phase field to below T_α , it transforms to ordered α_2^s that is supersaturated with aluminum and

contains stacking faults, from which γ plates nucleate and grow, resulting in alternate layers of α_2/γ lamellae. The γ plates formed via routes 1 and 2 contain thermal twins.

The third route is associated with the heat treatment of hot-worked two-phase alloys, during which α lamellae are formed upon heating in two possible fashions. In the first case, existing α_2 particles in the γ matrix disorder to α particles upon heating and grow into α plates, resulting in the high temperature lamellar structure $L(\gamma/\alpha)$. Upon cooling, $L(\gamma/\alpha)$ transforms to $L(\gamma/\alpha_2)$ by the α to α_2 ordering reaction. In the second case, upon heating, α precipitates out of the γ matrix and grows into α plates. The $L(\gamma/\alpha)$ lamellar structure transforms to $L(\gamma/\alpha_2)$ upon cooling. The lamellae that form via the third route usually consist of coarse α_2 plates ($>0.3 \mu\text{m}$) that are parallel to each other. Also, the γ plates all tend to have the same crystallographic orientation, as opposed to the thermally twinned γ plates that form via routes 1 and 2.

2.2.4.1 Control of the Lamellar Microstructure

The lamellar grain size, grain boundary morphology (smooth or interlocking), thickness, spacing and orientation of the lamellae are affected by alloy composition, heat treatment time, temperature, and cooling rates [11,53-64]. Increasing heat treatment time and/or temperature in the single-phase α field increases the lamellar grain size because of faster diffusion rates and the absence of other phases to inhibit the growth of α grains. Increasing the cooling rate (e.g. from furnace cool to air cool) results in smoother grain boundaries and a decrease in the thickness and spacing of the lamellae. In some cases, a

Widmanstätten-like lamellar morphology that has a “basket weave” appearance may also be created at high cooling rates, Figure 14(a). At very high cooling rates (e.g. water or ice quench), the formation of lamellae is suppressed and a massive transformation from α to γ occurs [58-64]. The resulting microstructure has a feathery appearance, Figure 14(b). The effects of these microstructures on mechanical properties have not been systematically addressed.

The fully lamellar microstructure is commonly found in cast materials. Although this microstructure has good fracture toughness (K_{IC} as high as 35 MPa $\sqrt{m}$) and creep resistance, its room temperature ductility is very poor (typically less than 1 % elongation) [15]. The latter has been attributed to a large grain size and compositional inhomogeneity in the microstructure [10,25]. It has been shown that improvement in room temperature ductility can be achieved by reducing the lamellar grain size [32]. However, this is difficult because of the rapid grain growth kinetics in the α single-phase field. Innovative techniques are required to refine the grain size of fully lamellar structures. Some of these techniques are described in the following sections.

2.2.4.2 Fine-grained Lamellar Microstructure

The preceding discussions indicate that the standard microstructures (near gamma, duplex, near fully lamellar, and coarse-grained fully lamellar) do not have a good mechanical property balance, and that a fine-grained lamellar microstructure may provide this desirable quality. However, because grain growth occurs rapidly in the single-phase α field, refining the grain size of the lamellar structure is very difficult by conventional heat

treatments. The results to date show that elaborate compositional modifications and innovative processing techniques are required to produce FGFL microstructures [9,11,16-18,65]. However, most of these methods are proprietary. The general approach of these techniques is presented below.

2.2.4.3 Compositional Modifications

The grain size of the fully lamellar microstructure in cast alloys can be reduced by the additions of boron (>0.8 at.%), silicon ($0.5-0.8$ at.%), and TiB_2 (0.8 vol.%) [11-15]. The latter method, developed by Martin Marietta Laboratories, is commonly referred to as the XDTM process. It involves in-situ formation of ≥ 0.8 volume % of TiB_2 particles, which are approximately $1\mu\text{m}$ in diameter and serve as nuclei for grain formation during solidification. A large number of these nuclei would therefore reduce the grain size of the material [12-14]. The resulting refinement is very uniform and quite independent of the size of the ingot. Figure 15 compares the microstructures of a cast alloy with and without 0.8 vol.% TiB_2 . Compared with their monolithic counterparts, the XDTM alloys have better room temperature tensile yield and ultimate strengths, but lower tensile elongation and fracture toughness [15]. The increase in tensile strength in the XDTM alloys is thought to be due to the refined grain size. The borides are not expected to act as dispersion strengthening agents. However, the flexibility in modifying the microstructure of XDTM processed alloys is limited because the TiB_2 particles retard morphological transformation.

For wrought alloys, the lamellar grain size can be refined by additions of B in the range of 0.2 to 0.3 at.% [11]. The effect is attributed to the presence of fine “lacy” Ti-borides that limit excessive grain growth during heat treatment in the single-phase α field. Examples of the effect of B additions on the grain size of a binary and a multicomponent alloy are shown in Figure 16.

Another approach that has been reported involves the additions of relatively large amounts of β stabilizing elements that expand the ($\alpha+\beta$) field in the phase diagram to lower temperatures [11]. Heat treatments can then be carried out in the expanded ($\alpha+\beta$) field during which the growth of α grains is limited by the presence of β phase. The α grains are then transformed into lamellar grains during subsequent heat treatment. Using this method, a lamellar grain size in the range of 100 to 200 μm has been reported for a Ti-46.5Al-2Cr-3Nb-0.2W alloy. This technique, however, requires careful composition modifications to avoid disproportionate amount of β phase in the ($\alpha+\beta$) field that can lead to compositional inhomogeneity and excessive grain refinement [11].

2.2.4.4 Heat Treatment

Very recently, it has been demonstrated that using a two-stage heat treatment, the as-cast coarse-grained lamellar microstructure of a Ti-46.6Al-2.3V-0.9Cr alloy can be transformed into a fine-grained lamellar structure with a grain size of 100 μm [16]. The first treatment involved annealing the as-cast lamellar microstructure in the $\alpha+\gamma$ two-phase field at 1200 °C for at least 50 hours. During annealing, the α plates spheriodized by continuous and discontinuous coarsening, resulting in a near-gamma structure. The second

treatment was conducted at above the T_{α} temperature to convert the near- γ structure into single-phase α . It was found that when the duration of the second treatment was limited to 7 minutes, a fine-grained lamellar structure with a grain size of 100 μm was developed upon cooling. However, because the initial near-gamma structure is produced by continuous and discontinuous coarsening mechanisms, which are affected by alloy composition and the morphology of the starting as-cast microstructure [66-69], it is expected that the effectiveness of this technique may vary depending on these parameters.

2.2.4.5 Thermomechanical Processing

It has been reported that a fine-grained lamellar microstructure can be obtained by hot extrusion at temperatures near or above the α transus temperature. The technique was applied to an ingot material [11] and prealloyed Ti-47Al-2Cr-2Nb P/M material [9,65]. In both cases, the lamellar microstructure produced had a colony/grain size in the range of 65 μm . Details of the composition and extrusion parameters for the cast material were not disclosed. However, it was noted that the lamellar grains were elongated in the extrusion direction. In the P/M material, extrusion was carried out at a ratio of 16:1 at a temperature above the α transus temperature. The resulting lamellar grains were oriented in a more random fashion. Although thermomechanical processing is effective to refine the lamellar grain size in wrought materials, it cannot be used to refine investment cast near net shape materials.

2.3 Mechanical Properties

The mechanical properties of γ -TiAl alloys are strongly influenced by their microstructures, which are in turn controlled by processing routes and chemical composition. The following sections review the effects of these factors on the tensile properties, fracture toughness, creep, fatigue crack initiation and propagation behaviour of γ -TiAl alloys. To provide a quantitative perspective for the discussion, a partial list of mechanical property requirements for several aero engine components is presented in Table 2 [70,71]. It can be noted that for compressor applications, a room temperature tensile ductility of 3%, yield strength in the range of 500-600 MPa, and a fracture toughness of 28 MPa $\sqrt{m}$ are required. For applications in which creep deformation is an important design consideration, the allowable limit for elongation ranges from 0.1 to 1% for a given time duration, depending on the applied stress and temperature level.

2.3.1 Effects of Composition, Elemental Additions and Impurities

One of the major shortcomings of γ -TiAl alloys is their lack of room temperature tensile ductility. During the initial stage of alloy development, it was found that an aluminum content in the range of 47 to 49% and a duplex microstructure provided the best room temperature ductility. As a result, this range of aluminum concentration has become the basic composition of multicomponent alloys.

The multicomponent alloys that have been investigated generally consist of Ti-(45-49 at.%)Al-(1-3%)X-(1-5%)Y-(0.1-1%)Z [34]. The elements in group X are Cr, Mn and/or V, which improve the ductility but reduce the oxidation resistance. The elements in group

Y include Nb, Ta, W and/or Mo, which have a solid-solution strengthening effect and improve the creep and oxidation resistance. The elements in group Z are C, N, B and Si. Small additions of C and N improve the creep properties, whereas B and Si refine the microstructure. The influence of various alloying elements on properties is summarised in Table 3 [20,21,34]. The compositional effect has been explained in terms of intrinsic changes in the crystal structure of the γ phase, reduction of the stacking fault energy, modification of the Ti-Al chemical bonding, shifts in the boundaries of the phase diagram, and/or microstructural effects such as lamellae thickness and spacing, the formation of minor phases that inhibit grain growth or the formation of other phases. However, there are many uncertainties because most of these effects have not been isolated and studied systematically. More detail on this subject is given in the following discussions.

Interstitial impurities such as oxygen, carbon and nitrogen are usually present in γ -TiAl alloys. Typical contents of these elements are in the range of 500-1000 ppm, 20-50 ppm, and 5-20 ppm, respectively. These impurities tend to increase strength and reduce ductility. However, there is evidence that if their concentrations are small, they may be absorbed by the α_2 phase and thus avoid a loss of ductility [20,72].

2.3.2 Tensile Properties

The tensile properties of γ -TiAl alloys are strongly dependent on alloy composition and microstructure, as well as on test conditions [11,20-22,72]. Among the standard microstructures, the fine-grained duplex microstructure generally has the best tensile ductility, whereas the reverse is true for the coarse-grained fully lamellar microstructure. It

is uncertain whether the difference is due to a microstructural or a grain size effect. This issue has been clarified recently when a fine-grained lamellar structure was developed and its properties were compared with those of standard microstructures [17,18]. γ -TiAl alloys often exhibit a drastic increase in ductility and a reduction in strength when the test temperature is elevated to above the temperature range of 600-800 °C, displaying a so-called brittle-to-ductile transition behaviour [73-81]. Under particular microstructural and test conditions (temperature and strain rate), some alloys also exhibit a superplastic deformation behaviour [30,82-86].

2.3.2.1 At Room Temperature

Aluminum content has a significant influence on the microstructure and tensile properties of γ -TiAl alloys. In single-phase alloys, decreasing aluminum concentration reduces the lattice tetragonality of the γ phase and consequently improves the ductility. For binary two-phase alloys either in cast or wrought condition, varying the aluminum content changes the morphology of the microstructure, resulting in different tensile properties. For cast alloys with a fully lamellar microstructure [5,87,88], when the aluminum content is below 47-48 at.%, the boundaries of the lamellar grains are smooth and straight. When the amount of aluminum is in the range of 47-48 at.%, the grain boundaries become wavy, whereas above this range, equiaxed γ grains begin to appear at the lamellar grain boundaries. The best combination of strength (≈ 600 MPa) and elongation ($\approx 2.5\%$) occurs when the aluminum content is approximately 48 at.%. Deviating from this composition results in a drastic reduction in these properties, Figure 17 [5]. A similar ductility value has

been cited for a cast alloy containing Ti-48Al-2Nb-2Cr that is heat treated to have a cast-duplex microstructure [15].

In wrought alloys that contain less than 46% aluminum, a brittle fully lamellar microstructure tends to form during processing, whereas above this range a single-phase gamma or a near-gamma microstructure predominates, Figure 18 [89,90]. On the other hand, alloys that contain 46-50% aluminum are inclined to have a duplex fine-grained microstructure that is significantly more ductile than the fully lamellar and the near-gamma microstructures.

The ductility of binary alloys can be improved by ternary additions of 1- 3 at.% Cr, Mn or V, Figure 19 [20.91-95]. However, this improvement is realised only in the duplex microstructure and not in the fully lamellar or the single-phase gamma microstructures, the reason for which is not clear. The ductilizing effects of Cr, Mn and V have been explained in terms of changes in the lattice parameters 'a', 'c', c/a ratio, and in the unit cell volume of the γ phase as a result of alloying element atoms occupying the Ti and/or the Al site. But these intrinsic factors are not the only conditions that control ductility because ternary additions of other elements, for example Nb and W, also bring about similar intrinsic changes, but yet reduce the ductility [20.21.72].

The tensile ductility of γ -TiAl alloys has been correlated with microstructural features including volume fractions of lamellar grains and γ grains, the morphology and volume fractions of α_2 and γ laths, and grain size [72]. Figure 20 [34] shows the tensile properties of a cast, forged and heat treated Ti-47Al-1Cr-1V-2.5Nb alloy with different microstructures and grain sizes. It can be noted that ductility is maximum when the

volume fraction of lamellar grains and γ grains are more or less equal, i.e. in the duplex microstructure condition. The coarse-grained fully lamellar microstructure in which γ grains are absent, exhibits higher strength but poor ductility. It is interesting to note that curve D in Figure 20, which represents a near-lamellar structure that contains predominantly lamellar grains with a grain size of 100 μm and a small amount of γ grains 20 μm in size, exhibits the best strength and ductility combination. More importantly, curves A, B and C clearly show that the tensile properties of the fully lamellar microstructure can be improved drastically by decreasing the grain size from 1500 μm to 300 μm . It was suggested that additional improvement may be possible by a further reduction in the lamellar grain size. This has been confirmed in recent studies in which fine-grained fully lamellar microstructures with a grain size in the range of 50-65 μm were obtained using novel thermomechanical processing and heat treatment procedures [9,11,16-18,65]. Figure 21 compares the properties of the fine-grained lamellar structure with those of the standard microstructures in a cast, forged and heat treated Ti-46.6Al-2.3V-0.9Cr alloy. The results show that when the grain size of the fully lamellar microstructure is reduced from 1200 μm to 52 μm , the ductility and strength increase drastically from ~ 380 MPa and 0.8% to ~ 520 MPa and 3.5% [17]. The fine-grained lamellar structure arguably has the best combination of strength and ductility. It can be observed that the ductility of the fine-grained lamellar structure falls in the upper range of tensile ductility values for fine-grained duplex microstructures, and that it meets the room temperature ductility requirement for compressor applications listed in Table 2.

It has been demonstrated that the tensile strength and ductility of gamma alloys depends mainly on grain size which is relatively insensitive to microstructural type, whereas the yield stress depends on both [11,18,25,96-97]. Figure 22 depicts the effect of grain size on tensile ductility and ultimate strength [18]. The microstructural conditions considered include coarse-grained and fine-grained fully lamellar, fine-grained duplex and near-gamma formations. It can be noted that all of the data fall along the same trend, regardless of microstructural state. However, this is not the case for yield strength. As shown in Figure 23, for grain size larger than $\sim 200 \mu\text{m}$, the fully lamellar microstructure exhibits higher yield strength. As grain size decreases, the difference in yield strength between the microstructures diminishes and all of the data appears to converge.

The yield stress dependence on grain size of a variety of alloys has also been correlated by the Hall-Petch relationship [11,17,25,96,97], an example is shown in Figure 24 [11]. It can be observed that the slope for the fully lamellar microstructure is relatively steep compared with that of the duplex or near-gamma microstructures, indicating its yield stress is more sensitive to grain size.

2.3.2.2 At Elevated Temperatures

In γ -TiAl alloys, a drastic increase in tensile ductility and attendant decreases in yield stress and fracture stress often occur when the temperature reaches the 'brittle-ductile transition temperature' (BDTT). This behaviour, shown schematically in Figure 25, has been observed in γ -TiAl alloys in various microstructural conditions [25,73-81]. The drastic change in tensile properties at temperatures above the BDTT is associated with

increased twinning and slip activity, as well as recrystallization induced by deformation. In general, the duplex microstructure has the lowest BDTT ($\approx 550\text{-}600\text{ }^{\circ}\text{C}$) and the near fully lamellar or fully lamellar structure has the highest value ($\approx 750\text{-}800\text{ }^{\circ}\text{C}$), whereas the BDTT of the single-phase gamma structure occurs between these extremes. It is not certain whether the variations in BDTT are due to the differences in grain size or phase distribution. Also, it has been shown that the presence of solid solute strengthening elements such as W impede deformation and consequently raise the BDTT [81].

2.3.2.3 Tensile Deformation Mechanisms

At room temperature, deformation in single-phase alloys occurs mainly by limited amounts of unit dislocation and super-dislocation motions. Twinning is generally not observed. In alloys with a duplex microstructure, deformation occurs predominantly in the γ grains by $\{111\}$ twinning and easy slip of $1/2[110]$ unit dislocations. Super-dislocations are essentially absent. The amount of deformation in the transformed lamellar grains is small. Most of the twinning occurs in the γ laths and very few deformation substructures are observed in the α_2 laths. Although extensive dislocations are present in the γ/α_2 lamellar interfaces, very few of them are transferred from the γ laths across to the α_2 laths. In alloys with a fully lamellar microstructure, very low dislocation activity and almost no twinning activity are observed [33,73-78].

The above observations had initially led to the belief that the lamellar structure in polycrystalline γ -TiAl alloys is intrinsically undeformable. But this has been shown to be not the case by tensile and compressive tests conducted on binary TiAl “polysynthetically

twinned” (PST) crystals that contain one set of γ and α_2 lamellae aligned in the same direction [21,98-104]. Some of the data are presented in Figure 26 [100], which shows that both yield stress and elongation depend strongly on the angle ' θ ' between the loading axis and the lamellae. When the loading axis is perpendicular or parallel to the lamellae ($\theta = 90^\circ$ or 0°), tensile elongation is low and strength is high. In these orientations, a “hard” mode of deformation occurs in which shear deformation proceeds predominantly in γ laths on $\{111\}$ slip planes that are oriented across the lamellae. The slip deformation, however, is obstructed by the lamellae boundaries and the α_2 laths, resulting in high strength and low ductility. For intermediate orientations, at $\theta = 30^\circ$ to 60° for example, a “soft” mode of deformation occurs in which shear proceeds in γ lamellae on $\{111\}$ slip planes that are parallel to the lamellar boundaries. In this mode, the lamellae boundaries and the α_2 laths do not directly impede the deformation, resulting in lower strength and higher ductility. The orientation effect on tensile properties has been further demonstrated by test results obtained from a bicrystal that consisted of two lamellar grains, one above the other Figure 27 [101]. The upper grain contained lamellae that were aligned for the “easy” mode of deformation ($\theta = 40-60^\circ$), and the lower grain contained lamellae that were aligned for the “hard” mode of deformation ($\theta = 75-80^\circ$). A tensile elongation of 4% was obtained in the bicrystal. However, most of the deformation occurred in the upper grain in the “easy” mode of deformation. As deformation proceeded, strain discontinuity between the two grains became larger. Finally, failure was initiated at a γ/α_2 lamellar interface near the grain boundary.

The above observations are significant because they show the lamellar structure can be highly deformable, and that the poor ductility of polycrystalline γ -TiAl alloys with a fully lamellar microstructure can be due to strain discontinuity between adjacent lamellar grains with different lamellae orientations. It is therefore expected that reducing the size of the lamellar grains would reduce strain discontinuity and improve ductility. This is indeed consistent with the tensile test data for the fine-grained lamellar microstructure shown in Figures 20 and 21.

2.3.3 Fracture Toughness

It has been observed that the presence of lamellar grains is advantageous to the fracture resistance of γ -TiAl alloys [18,105-113], and that for a given microstructure type and within a certain limit, fracture toughness increases with lamellar grain size. Figure 28 [18,105,106], volume fraction of the lamellar grains. Figures 29 and 30 [88,113], and lamellae spacing, Figure 31 [105]. Among the standard microstructures, the fully lamellar structure has the highest fracture toughness ($K_{IC} \approx 30 \text{ MPa}\sqrt{\text{m}}$), followed by the duplex and then the single-phase γ structure. The relationship between microstructural type and fracture toughness is therefore the inverse of that between microstructure and tensile ductility. Recently, it has been shown that the inverse relationship can be reduced by refining the lamellar grain size, Figure 28 [18,105].

Typical fracture modes of different microstructures are schematically presented in Figure 30 [88]. In duplex and single-phase γ microstructures, the volume fraction of lamellar grains is low and the fracture mode is predominated by grain boundary decohesion

and cleavage fracture of the equiaxed γ grains. The superior fracture resistance of the fully lamellar microstructure is due to the presence of α_2/γ lamellae, which imparts a composite-like fracture behaviour to the material and increases its propensity for crack branching. The crack initiation process is characterised by the formation of non-coplanar cracks at lamellae interfaces, and crack propagation involves translamellar fracture, and deflection and arrest of advancing cracks by the α_2/γ lamellae and by the grain boundaries. These observations clearly suggest that lamellae orientation plays a role in the fracture resistance of materials containing lamellar microstructure. However, no systematic study has been reported in this regard. In the fully lamellar condition, a serrated grain boundary is more beneficial for fracture resistance than a smooth grain boundary because it leads to tortuous intergranular crack paths, Figure 30b. It has also been shown that the fracture toughness of the fully lamellar structure increases with decreasing lamellae spacing, Figure 31 [105,113], although the benefit appears to diminish when the spacing is less than 1-2 μm . It is important to note from Figure 31 that when the grain size of the fully lamellar microstructure is reduced by two orders of magnitude (from 1400 μm to 60 μm), the fracture toughness is reduced by a much smaller extent (from $\sim 30 \text{ MPa}\sqrt{\text{m}}$ to $\sim 20 \text{ MPa}\sqrt{\text{m}}$). This observation is supported by recent studies on fine-grained lamellar microstructures [9, 17, 18], which showed the K_{IC} for lamellar grain sizes in the range of 45-65 μm was $\sim 22 \text{ MPa}\sqrt{\text{m}}$, compared with $\sim 28 \text{ MPa}\sqrt{\text{m}}$ for a lamellar grain size of 500 μm , Figure 32 [18]. These results, along with the tensile test results presented in Figure 21, indicate a fine-grained lamellar microstructure can provide much better strength and ductility than their coarse-grained counterpart without a significant loss in fracture toughness.

2.3.4 Creep

The creep properties of γ -TiAl alloys are influenced by microstructural condition, alloy composition, and test conditions. The fully and near fully lamellar microstructures have significantly better creep resistance than those with the duplex and the single-phase microstructures. A number of parameters, including grain size, volume fraction of lamellar grains and equiaxed γ grains, lamellar grain boundary morphology, and lamellae spacing, play an important role in creep resistance. The addition of certain alloying elements can also improve creep resistance by solid solution strengthening, or the formation of minor phases that inhibit deformation and dynamic recrystallization during creep. However, because these parameters cannot be easily isolated from one another and studied systematically, their individual effects on the creep resistance of multiphase TiAl alloys are not fully understood [114]. The following sections provide more detail on these effects.

2.3.4.1 Microstructural Effects

The effects of microstructure and volume fraction of lamellar grains on creep resistance are shown in Figures 33 and 34 [21, 25, 33, 43, 44], respectively. It can be observed that the presence of lamellar structure reduces creep strain rate and increases rupture life. However, the volume fraction of lamellar grains required to be effective in reducing creep rate may vary from approximately 50 % to 80 % [114]. This apparent inconsistency may be due to a concurrent increase in grain size with volume fraction of lamellar grains, as well as differences in lamellar grain boundary morphology, and the distribution of γ grains at the lamellar grain boundaries. With regard to the latter, the

presence of γ grains at lamellar grain boundaries is generally considered as undesirable because it increases the propensity for dynamic recrystallization and promotes grain boundary sliding, which hastens the onset of tertiary creep. However, it has been reported that the superior creep performance of the fully lamellar microstructure over that of the duplex microstructure decreases with decreasing temperature, Figure 35 [115]. At temperatures below 630 °C, the duplex microstructure exhibits higher stress rupture strength than the fully lamellar structure. It is thought that at low temperatures, the lamellar structure behaves in a brittle manner, and that grain boundary sliding and diffusional creep are not operative to diminish the creep life of fine-grained duplex materials [115].

In the fully lamellar condition, a large grain size and an interlocking grain boundary morphology are considered to be beneficial for creep resistance because they reduce diffusional creep and grain boundary sliding. In addition, the presence of secondary Widmanstätten-like lamellar colonies inside the lamellar grains can be advantageous in reducing creep deformation by a ligament bridging mechanism [116,117]. It is also believed that a fine lamellae spacing (less than $\approx 400\text{-}500$ nm) is beneficial for creep resistance because the α_2 and γ lamellae interfaces act as obstacles to dislocation motion [43,65,116-121]. Recently, it has been reported that a fine-grained lamellar microstructure with a grain size of 65 μm , a lamellae spacing of 100 nm, and interlocking grain boundaries, exhibits lower strain rates than those of the coarse-grained lamellar structures, Figure 36 [65]. These results demonstrate that a better balance of creep and tensile properties for γ -TiAl alloys can be achieved by refining the grain size and modifying the lamellar microstructure. However, the effects of lamellar grain size and lamellae spacing on

creep rate are not yet fully understood. In recent studies, it has been observed that minimum creep rates were insensitive to grain sizes in the range of 210 to 1200 μm , Figure 37 [116,117], and lamellae spacing in the range of 75 to 300 nm.

2.3.4.2 Effects of Alloy Additions on Creep Properties

It has been shown that additions of Nb, Ta, Mo, W and Si increase the creep resistance of binary γ -TiAl alloys by solid solution strengthening and/or modifying the microstructure [20,121-125]. In this regard, W and Mo are effective in promoting the formation of β phase particles along lamellae interfaces, rendering the lamellae more resistant to dynamic recrystallization and spheroidization, and consequently delaying the onset of tertiary creep [117,123,124]. In a similar manner, additions of 0.5-1% Si drastically improve the creep rupture life of binary alloys by precipitating fine Ti_5Si_3 particles on γ/α_2 lamellae interfaces during creep deformation, which prevent the lamellae from dynamic recrystallization. However, when the Si concentration exceeds 1%, the volume fraction of lamellar grains is reduced, resulting in a shorter creep rupture life. Interstitial addition of C and N can improve creep life by lowering the creep rates. The mechanisms responsible for the improvement of creep properties by C and N interstitial additions are not fully understood. It is thought that they may be related to a solid solution strengthening effect, and to the precipitation of Ti_2AlC and Ti_2AlN particles that impede dislocation movements during creep deformation [20,114,125,126]. It has been observed that C additions are more beneficial for the duplex and single-phase microstructures than for the lamellar microstructure [126]. A possible explanation is that other microstructural

parameters, such as lamellae spacing, are more influential than interstitial addition effects on the creep behaviour of the lamellar structure.

2.3.4.3 Creep Deformation Mechanisms

The creep behaviour of multiphase TiAl alloys may consist of three stages: primary, secondary and tertiary. The primary creep behaviour has not been studied extensively, however, there is evidence that primary creep strain increases with the volume fraction of lamellar grains. This effect has been attributed to the presence of numerous lamellae interfaces [127] and the high density of dislocations initially present at these interfaces, which may readily accommodate initial deformation. The secondary stage of creep receives the most attention because it constitutes the major portion of the total creep life. Two types of behaviour have been reported during secondary creep, a steady-state behaviour in which the creep rate remains relatively constant for a long period of time before the rate increases, or a “pseudo steady-state” behaviour in which the rate of creep deformation decreases to a minimum and then increases [114]. During tertiary creep, the rate of deformation accelerates, which ultimately leads to instability and rupture of the specimen. The following power law creep equation is often used to correlate creep rate with applied stress and temperature:

$$\dot{\epsilon} = A\sigma^n \exp^{-Q/RT} \quad (\text{Eq. 1})$$

In this equation, $\dot{\epsilon}$ is the creep rate; “A” is a microstructure dependent constant that represents parameters including stacking fault energy, dislocation configuration, grain size, and the type and dispersion of second phase precipitates; “n” is the apparent stress

exponent; “Q” is the apparent activation energy; “R” is the universal gas constant; and “T” is the absolute temperature. In general, the value of n increases with applied stress. $n \approx 1$ is associated with a test condition under which the creep process is controlled by diffusion, whereas a value of $n \approx 4.5$ and above indicates the creep process is controlled by dislocation climb and glide, respectively [114]. The majority of the creep data in the literature for nearly and fully lamellar microstructures shows the value of Q in the range of 350-400 kJmol⁻¹, which is more than the activation energy of 292 kJmol⁻¹ for self diffusion of Ti in TiAl. This suggests that diffusion may not be the controlling deformation mechanism [123]. In addition, the values of “ n ” for a majority of the data fall in the range of 4 to 8 [114,118-121,126-131], which indicates creep deformation is predominantly controlled by dislocation related processes. This is consistent with the type of deformation substructure observed in specimens that have been tested in the minimum creep regime, which consists of some twinning in the γ laths, dislocation tangles and well developed subgrain boundaries from $1/2 \langle 110 \rangle$ dislocation arrays in the γ grains [130]. In the γ lamellae, $1/2 \langle 110 \rangle$ dislocation structures and microtwins also develop. A similar dislocation morphology is also observed in the fully lamellar structure. As a result of the above observations, a finer lamellae spacing and therefore a shorter slip distance, is generally considered to be beneficial in reducing minimum creep rate because the lamellar interfaces limit dislocation movement [65,114,128].

During the tertiary creep of multiphase TiAl alloys, microstructural instabilities often occur [116-121,129,130]. In duplex and near fully lamellar microstructures, dynamic recrystallization takes place in the single-phase γ grains at the lamellar grain boundaries,

creating regions of highly localised deformation that eventually become crack initiation sites. In fully lamellar microstructures, tertiary creep is controlled by the nucleation and growth of voids at the grain boundaries [118,127], or by intragranular deformation mechanisms that include the break up of the lamellae and spheroidization of α_2 laths [116,118,120,121,130]. The latter process can occur in two modes, depending on the orientation of lamellar grains relative to the loading direction [116,121,130]. In the grains that are oriented with their lamellae parallel to the loading direction, a “hard” mode of deformation takes place in which a network of shear bands are formed at approximately 45 degrees from the loading axis. The shear bands contain fine recrystallized grains which are believed to be the result of dynamic recrystallization. When the lamellae are perpendicular to the loading axis, bending and distortion of the lamellae take place. The hard and soft modes of deformation have been described earlier in the section on tensile deformation mechanisms.

2.3.5 Fatigue Crack Initiation and Propagation

The effects of microstructure on the fatigue strength of near- γ TiAl alloys have been investigated to a limited extent [21,43,70,132-139]. It appears that at test temperatures below the ductile to brittle transition temperature (BDTT) range, the duplex microstructure has the same, or better fatigue resistance than the fully lamellar microstructure, whereas at temperatures above the BDTT, the fully lamellar structure generally has longer high cycle fatigue life, Table 4 and Figure 38 [43]. The explanation for this has not been studied systematically. It may be because at high temperatures, the fully lamellar microstructure

behaves in a less brittle manner, and the coarse grain size and interlocking grain boundaries are more effective in resisting fracture than the fine-grained duplex structure. It is noteworthy from Table 4 that the fatigue strength of a refined fully lamellar microstructure (alloy K5), which has a grain size of 300 μm , exhibits one of the highest fatigue strengths compared to the other microstructures. It can also be observed that the ratio of fatigue strength to yield strength for some of the alloys approach or exceed unity (e.g. alloy K5). This is thought to be partly due to cyclic work hardening and strain rate effects that occur during high frequency fatigue testing [21,43,132,136,138].

Fatigue crack propagation rate data for near- γ TiAl alloys are scarce in the open literature. Available test results show that fatigue crack growth in these materials is much higher than it is for superalloys even after density adjustment. The steady-state crack growth region between the threshold stress intensity factor (below which fatigue crack growth is negligible) and the critical threshold stress intensity factor is very limited, indicating that these materials are not damage tolerant [70,139-145]. It is generally observed that the lamellar microstructure has much higher fatigue crack growth resistance than that of the duplex microstructure, Figures 39 [70,145]. In the duplex microstructure, fracture initiates in the equiaxed gamma grains by transgranular cleavage at room temperature and intergranular decohesion at elevated temperatures. In the lamellar microstructure however, crack growth is highly dependent on the orientation of the lamellae with respect to the crack propagation direction, especially in the low stress intensity factor region. It has been observed that fatigue crack growth rate is slower when the crack is perpendicular to the lamellar interfaces, Figure 40 [142]. This phenomenon

has been attributed to the formation of shear ligaments ahead of the crack tip that deflect and delay crack growth [140-145]. More importantly, it has also been shown that under certain conditions, crack arrest can occur at lamellar grain boundaries that separate grains with distinctly different lamellae orientations. An example is shown in Figure 41 [144], which shows crack growth is stopped at the grain boundary, and remains arrested until the applied stress intensity factor is increased from 8 MPa $\sqrt{\text{m}}$ to 13 MPa $\sqrt{\text{m}}$. These results suggest that fatigue crack growth resistance in fully lamellar structure may be further improved by increasing the number of grain boundaries by refining the grain size. In fact, a recent review cited threshold stress intensity factors of 15-20 MPa $\sqrt{\text{m}}$ for refined fully lamellar materials, compared with 5-10 MPa $\sqrt{\text{m}}$ for coarse-grained lamellar materials [43].

2.4 Summary

A number of important conclusions can be extracted from the preceding literature review:

1. There are four “standard” microstructures in two-phase γ -TiAl alloys: a) near-gamma, b) fine-grained duplex, c) near fully lamellar, and c) coarse-grained lamellar.
2. These standard microstructures do not exhibit a good mechanical property balance. The near-gamma and duplex microstructures have better room temperature tensile strength and ductility but poor creep strength, fatigue crack growth and fracture resistance, whereas the reverse is true for materials that have near fully lamellar or coarse-grained fully lamellar microstructures.

3. There is strong evidence that a fine-grained fully lamellar microstructure can significantly improve the balance in tensile properties, fracture toughness, fatigue strength, fatigue crack growth resistance and creep properties of γ -TiAl alloys.
4. The desirable features in the lamellar structure include: a fine lamellar grain size in the range of 50-100 μm , an interlocking grain boundary morphology, a lamellae spacing less than approximately 0.1 μm , and the presence of minor phase particles that stabilize the lamellar structure and inhibit deformation at high temperatures. These features are influenced by chemical composition, thermomechanical processing and heat treatment parameters.
5. Because of the nature of phase transformations in near-gamma alloys, it is difficult to refine the lamellar grain by conventional heat treatment. To date, fine-grained fully lamellar microstructures have been produced in cast and P/M materials by combination of compositional modifications, hot work and heat treatment. However, there has been no report that this microstructure can be produced in P/M materials using heat treatment only.
6. The objective of this thesis is to develop heat treatment techniques to produce FGFL microstructures in a P/M material. It is hoped that the knowledge and techniques developed herein can be extended to cast or wrought materials.

2.5 Thesis Approach in Brief

The review of the phase diagram and microstructures presented in sections 2.1 and 2.2 shows that when heat treatments are conducted slightly below the α transus

temperature, a microstructure that consists of somewhat refined lamellar grains and equiaxed γ grains is produced. The heat treatment procedure developed in this thesis was based on the phase transformation kinetics in this regime. A series of heat treatments were first conducted to determine the α transus temperature for the material, and the effects of heat treatment time and cooling rates on microstructure. Additional heat treatments were then carried out at and near the α transus temperature to determine the $\gamma \rightarrow \alpha$ transformation kinetics. On the basis of these results, a procedure that consists of heat treatment in the α transus temperature region in a cyclic manner was developed to produce fine-grained fully lamellar microstructures. A range of lamellar grain sizes were obtained by adjusting the number of heat treatment cycles and temperature. The room temperature tensile and the high temperature creep properties of the FGFL microstructure were compared with those of the duplex and the fully lamellar structures to show that the FGFL structure exhibited much better balance in properties.

CHAPTER 3

MATERIAL AND EXPERIMENTAL PROCEDURE

3.1 Material

The starting material is an induction melted and gas atomized prealloyed powder metallurgy material that has a nominal composition of Ti-47.5Al-3Cr (at.%). The material was supplied by Crucible Compaction Metals and delivered in hermetically sealed containers to minimize contamination. Results of chemical analysis and size distribution measurements of the powder are presented in Table 4 and Figure 42, respectively [146]. A certificate of test for the powder is attached in the Appendix.

3.2 Experimental Procedure

The procedure to produce heat treatment samples and mechanical test specimens from prealloyed powder involves the following steps: 1) canning of the powder material; 2) consolidating the canned powder by hot isostatic pressing; and 3) encapsulating specimen blanks in quartz tubes for heat treatment. Figure 43 shows the product form at various stages in the process. Details of each of these steps are described below.

3.2.1 Canning of the Powder Material

Either commercially pure (99.9%) titanium or 304 stainless steel tubes were used for canning the powder. Stainless steel cans were used only for consolidation temperatures below 1200 °C because above this temperature, the material did not have

sufficient strength to maintain straightness. The outside diameters of the tubes ranged from 16 mm to 19 mm (5/8 in to 3/4 in) and the wall thickness ranged from 0.5 mm to 0.8 mm (0.02 in to 0.032 in).

The tubes were cut to approximately 36 cm in length and then ultrasonically cleaned for at least 10 minutes in petroleum ether to remove contamination on the inside surface. After cleaning, one end of the tube was clamped to close it using a hydraulically operated crimping tool, Figure 44, and then sealed by tungsten inert gas (TIG) welding. The sealed end was checked for leakage by evacuating the can for several hours. The vacuum system consists of a roughing pump and a diffusion pump, Figure 45; the system allows three cans to be evacuated simultaneously. A vacuum in the range of 8×10^{-6} mbar to 1×10^{-5} mbar was typically achieved in 5 to 6 hours when there was no leak in the weld.

The cans were then transferred to a hermetically sealed glove chamber, Figure 46, for filling. The chamber was purged with argon three times before filling to avoid contamination of the powder during the process. The cans were filled with powder typically to a height of 15 cm. The open end was then packed with glass wool to prevent the powder from entering the vacuum system during “degassing”, and plugged temporarily with rubber stoppers to avoid contamination after the cans were transferred out of the chamber. The stoppers were removed just before the cans were connected to the vacuum system for degassing, Figure 45. Degassing was initially conducted at room temperature until a vacuum of 1×10^{-5} mbar, or better, was reached. The cans were then heated in a vertical furnace, Figure 45(d), that had been preheated to 350 °C to increase

the gas pressure inside the cans to facilitate further degassing. This process was continued until a vacuum of approximately 8×10^{-6} mbar was reached. The cans were then allowed to cool. With the vacuum system on, each can was crimped progressively from the region immediately above the powder towards the open end to prepare the can for sealing, Figure 44. For titanium cans, the area to be crimped was preheated with an oxyacetylene torch to soften the material and thereby avoid the formation of cracks during crimping. This step was not necessary for stainless steel cans because of their high room temperature ductility. Finally, while maintaining the crimping pressure, the can was disconnected from the vacuum system and a cut was made above the crimped area. The cut end was subsequently sealed by TIG welding, after which the crimping pressure was released, and the weld was checked for cracks under an optical microscope before the can was subjected to consolidation.

3.2.2 Consolidation

The powder was consolidated by hot isostatic pressing (HIP) in a Autoclave Engineers press filled with argon. The process involved simultaneously applying heat and isostatic pressure to sinter the powder particles together. Figure 47 shows a schematic representation of the hot isostatic press. The press has a maximum operating temperature and pressure of 2000 °C and 207 MPa, respectively. The maximum heating rate for processing temperatures in the range of 1000-1300°C is typically 700 °C per hour. The processing temperature and pressure can be controlled within ± 10 °C and ± 2

MPa, respectively. The cooling rate can be controlled to any rate slower than a furnace cooling rate of approximately 750 °C per hour.

The cans were placed vertically inside an alumina crucible in the heating chamber of the press. The crucible was 15 cm in diameter and 30 cm in depth, it was filled with silica particles to provide support for the cans. Thermocouples were placed among the cans and in different regions of the crucible to control and monitor the processing temperature. Before each run, the chamber was purged three times with high purity argon to remove atmospheric air, and then pressurized with argon to 55 MPa (8 ksi). The pressure for consolidation was primarily provided by the heating cycle. A HIP cycle that consisted of 1250 °C/200 MPa /2 hours/furnace cool was used to produce compacts with > 99.9% full density [50,51]. A typical thermal history for this HIP cycle is shown in Figure 48. Typically six cans were consolidated in each HIP run. After HIP, samples were taken from the compact and examined metallographically to determine if the powders were fully consolidated, and that the microstructure obtained was consistent with expectation.

3.2.3 Encapsulation for Heat Treatment

The casing of the consolidated compact was removed by turning in a lathe to produce specimen blanks, Figure 43(e), approximately 1 cm in diameter for heat treatment. The heat treatment and mechanical test samples were approximately 1 cm and 6.5 cm in length, respectively. The samples were encapsulated in argon filled quartz tubes (25 mm outside diameter and 1.5 mm thickness) to prevent oxidation during heat

treatment. The quartz tubes were cleaned in ethyl alcohol, rinsed in distilled water and dried in an oven at 250 °C before use. To avoid the diffusion of Si from the quartz tube into the specimen at high temperatures, direct contact between the sample and the quartz tube was prevented by either wrapping the entire sample in pure tantalum foil, 0.125 mm in thickness, or wrapping the ends with tantalum wire 2 mm in diameter, Figure 43(e,f). The tubes were evacuated and purged five times with argon, and finally back filled with 26 kPa of argon before they were sealed. This internal pressure prevented the tube from collapsing as the quartz tube began to soften in the temperature range of 1300 to 1400 °C.

3.2.4 Heat Treatment

All of the heat treatments were conducted in a conventional radiation furnace using the internal thermocouple as control. The thermal gradient in the furnace was measured to locate the hot zone where the temperature gradient was a minimum. Temperature differences along the length of the sample were found to be within ± 2 °C. Samples were always placed within this region to minimize error in the expected processing temperature. Before each heat treatment, the furnace was allowed to stabilize at the set point temperature for at least two hours, and an external thermocouple was placed beside the specimen to monitor the hot zone temperature.

A majority of the heat treatment temperatures used in the study fell in the range of 1300 to 1360 °C. Within this temperature range, several heating and cooling rates were used to study their effects on microstructure. They include furnace heating rates of 6 °C/sec (“uninsulated furnace heat”) and 0.3 °C/sec (“insulated furnace heat”), a

furnace cooling rate of 0.3 °C/sec (“furnace cool”), air cooling rates of 4 °C/sec (“insulated fan air cool”) and 8 °C/sec (“uninsulated fan air cool”). Water quench was also used in a number of heat treatment situations. An apparatus was devised to estimate the heating and cooling rates of the encapsulated specimen during heat treatment. It consisted of a mechanical test specimen blank with a center hole drilled to a depth approximately half of its length. A thermocouple was embedded at the bottom of the blind hole and the instrumented specimen was wrapped in tantalum foil and placed at the bottom of a long quartz tube. The tube was then filled with argon and plugged with a rubber stopper. The heating and cooling rates measured by this apparatus are shown in Figures 49 and 50. The rate of cooling during water quench was not measured.

3.2.5 Metallography

Metallographical examinations were conducted using optical, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. For optical and SEM examinations, the specimens were polished electrolytically under the following conditions:

Electrolyte: methanol + ethylene glycol + perchloric acid in a volumetric ratio of 10:6:1

Voltage and current: 30 volts dc, 0.5 to 1 ampere; the specimen being the anode

Temperature and time : -25 °C to -30 °C for approximately 3 minutes

As-polished specimens were examined for the presence of porosity using an optical microscope, and for differentiation of phases in the SEM in the backscattered mode. To

reveal other microstructural features of interest such as grain size and phase distribution. the specimens were etched in a solution of glycerol, distilled water, nitric acid and hydrofluoric acid in a volumetric ratio of 25:12:12:1. Grain size measurements were made using the intercept procedure described in ASTM E 112-88, whereas phase distribution was estimated in an image analyzer under the assumption that area fractions were representative of volume fractions. A minimum of three locations in the specimen were used in each area analysis.

For TEM examination, cylindrical rods approximately 3 mm in diameter and 0.25 mm in thickness were machined from parent specimens. The foils were twin jet polished under the following conditions:

Jet polish solution: 64% methanol, 31% butanol, 5% perchloric acid in volume

Voltage/current: 13-15 vdc /20-30 ma.

Temperature and time : -45 °C to -50 °C for approximately 3 minutes

3.2.6 Mechanical Testing

Tensile tests and creep tests were carried out to compare the mechanical properties of different microstructures. For each of the microstructures, one tensile test and up to two creep tests were conducted. A stabilization heat treatment consisting of 950 °C/ 48 hours/ furnace cool was applied to all of the specimen blanks. Test samples were manufactured from heat treated blanks in accordance with the procedure and dimensions specified in Figure 51. Room temperature tensile tests were performed in a closed loop servohydraulic test frame under strain control at an engineering strain rate of

$1 \times 10^{-3} \text{ sec}^{-1}$. The error in stress and strain measurements were estimated to be $\pm 8 \text{ MPa}$ and $\pm 0.02 \%$, respectively. The typical setup for tensile test is shown in Figure 52.

Creep tests were performed at $760 \text{ }^{\circ}\text{C}$ under an initial applied stress of 240 MPa in a constant-load creep frame, Figure 53. The specimen was heated by a three-zone radiation furnace. The test temperature in the gage length was controlled to within $\pm 2 \text{ }^{\circ}\text{C}$ of the set point value. Three external thermocouples were attached to the gage length and grip sections of the specimen to monitor test temperature throughout the test. A linear variable differential transducer was attached to the shoulders of the specimen to measure tensile elongation. The maximum error of the transducer due to drifting was estimated to be less than 1.5% of the measured value. Specimen elongation and temperature were recorded by a multiple channel data recorder. The frequency of data acquisition ranged from fractions of a minute at the beginning of the test to several hours, depending on the creep rate.

After mechanical testing, the specimens were examined metallographically using optical, SEM and TEM techniques to characterize the failure mode.

CHAPTER 4

EXPERIMENTAL RESULTS

The review presented in sections 2.1 and 2.2 shows that when heat treatments are conducted slightly below the alpha transus temperature, T_α , microstructures that consist of somewhat refined lamellar grains and equiaxed γ grains can be produced. The cyclic heat treatment procedure developed in this thesis was based on the phase transformation kinetics in this regime. A series of heat treatments were first conducted to investigate the effects of heat treatment temperature, time, and cooling rate on microstructure, and to estimate the alpha transus temperature. On the basis of these observations, heat treatment parameters were selected to produce standard microstructures (duplex and fully lamellar) and fine-grained lamellar microstructures for mechanical property evaluations.

4.1 As-HIP Microstructure

After HIPing at 1250 °C and 200 MPa for 2 hours, the material appeared fully consolidated and porosity was rarely observed. When porosity was present, image analysis showed that it constituted less than 0.01% of the area examined. These results are consistent with those reported earlier in references [50,51].

The as-HIP microstructure is shown in an optical photograph and a SEM backscattered electronic image (BEI) in Figure 54(a) and 54(b), respectively. The structure consists of equiaxed γ grains with diameters typically ranging from 10 to 20 μm , however, large grains with size $\approx 100 \mu\text{m}$ are occasionally present, Figure 54(a). In BEI,

the brightness of a phase is proportional to the concentration and atomic weight of its constituent elements. Hence, in Figure 54(b), the α_2 -Ti₃Al and β_2 phases, which contain higher concentrations of the heavier elements Ti and Cr, appear brighter than the γ phase, which contains primarily Ti and Al (the atomic weights of Cr, Ti and Al are 24, 22 and 13, respectively). Qualitative SEM energy dispersive spectroscopy analysis of the near-white particles (denoted by arrows in Figure 54(b)) show that they are rich in Cr, Figure 54(c), indicating that these are β_2 phase particles [28, 29, 146]. The α_2 -Ti₃Al and β_2 phase particles are present primarily on the γ grain boundaries. Image analysis shows that the area fraction of grain boundary α_2 and β_2 phase particles is in the order of 5 %.

4.2 Microstructures after Static Heat Treatments

4.2.1 Effect of Temperature

As-HIP material was heat treated isothermally at temperatures ranging from 1300 °C to 1400 °C. The samples were heated at a rate of approximately 1°C/sec from approximately 800 °C to the target temperature, held at this level for two hours, followed by air cool at a rate of 8 °C/sec.

The microstructures and corresponding volume fractions of γ grains (V_{γ_g}) obtained are shown in Figures 55 and 56, and Table 6. At temperatures between 1300 °C to 1330 °C, fine-grained duplex microstructures consisting of equiaxed γ grains (~10 μ m in size) and lamellar grains (~30 μ m) were produced. As the heat temperature was increased, the size and volume fraction of lamellar grains increased, whereas the opposite occurred for γ grains. Heat treating at 1340 °C and 1345 °C resulted in near fully

lamellar microstructures consisting of large lamellar grains approximately 300 μm in size, and low volume fraction of equiaxed γ grains that were located predominantly at the lamellar grain boundaries. These observations are consistent with those reviewed in Chapter 2, section 2.2.

It is noteworthy that in the sample that was heat treated at 1345 $^{\circ}\text{C}$, a feather-like structure appeared, mostly at the lamellar grain boundaries (L_{gb}), Figure 55(b). This type of microstructure is considered to be an incipient form of a “massively transformed” gamma phase, γ_{m} , which has been associated with a diffusionless $\alpha \rightarrow \gamma$ during relatively fast cooling (e.g. air cool, water quench) from the α single-phase field, or during very rapid cooling ($> \sim 100$ $^{\circ}\text{C}/\text{sec}$) from the $\alpha + \gamma$ field [52-61]. (This subject is discussed further in 4.2.3.) Therefore, the appearance of feathery incipient γ_{m} in the 1345 $^{\circ}\text{C}$ air cooled sample suggests that this temperature may be above T_{α} . Increasing the heat treatment temperature further to 1350 and 1360 $^{\circ}\text{C}$ produced predominantly feathery γ_{m} microstructures that contained occasional lamellae. When the heat treatment was conducted at 1400 $^{\circ}\text{C}$, the microstructure was entirely feathery γ_{m} with poorly defined grain boundaries.

The observations above suggest the value of T_{α} lies between 1340 $^{\circ}\text{C}$ and 1345 $^{\circ}\text{C}$. This value needed verification because some of the transformations may not have been completed in two hours, especially at the lower temperatures. Also, one of the standard microstructures of interest, the fully lamellar structure, was not achieved by simply increasing the heat treatment temperature above 1340 $^{\circ}\text{C}$ followed by air cool at a rate of 8 $^{\circ}\text{C}/\text{sec}$.

4.2.2 Effect of Time

Isothermal heat treatments with periods of up to 100 hours were conducted at 1320 °C, 1330 °C, 1335 °C, 1340 °C and 1345 °C, followed by air cooled at 8 °C/sec, to estimate the value of T_α . It was assumed that a duration of 100 hours would allow the $\alpha_2 + \gamma \rightarrow \alpha + \gamma \rightarrow \alpha$ transformations to practically complete at these temperatures. After the value of T_α was determined, additional heat treatments with periods of 10, 30 and 60 minutes were carried out at temperatures slightly below and above T_α to determine the grain growth and $\gamma \rightarrow \alpha$ transformation kinetics in this regime.

4.2.2.1 Long-Time Heat Treatments

Figures 57 to 60 depict the microstructures of samples that were heat treated for 2 to 100 hours at 1320, 1330, 1335 and 1340 °C. The lamellar grain size (L_{gs}), γ grain size (γ_{gs}), and γ grain volume fraction ($V\gamma_g$) in these microstructures with respect to time are plotted in Figure 61 and summarized in Table 6(a). In all cases, when heat treatment time increased, L_{gs} and γ_{gs} increased, but $V\gamma_g$ decreased. The lamellar grain size L_{gs} can be correlated empirically with heat treatment time “t” in the form of a power function:

$$L_{gs} = kt^n \quad (\text{Eq. 2})$$

in which k is a constant and n represents the slope of the lamellar grain growth trend lines in Figure 61. The values for the constants k and n are recorded in Table 6b. It can be observed that although the scatter of the values of “n” is rather large for temperatures below T_α at 1320-1340 °C, it appears that n tends to increase with heat treatment temperature. A similar trend also exists at temperatures above T_α at 1350-1400 °C. The

increase in n with heat treatment temperature is consistent with a diffusion activated grain growth mechanism.

After 100 hours at 1340 °C, the nearly lamellar microstructure was characterized by an equiaxed morphology with smooth grain boundaries exhibiting a large number of ~120° dihedral angles, Figure 60, suggesting that the microstructure was close to equilibrium. In contrast, samples that were treated at lower temperatures after 100 hours did not exhibit these features, indicating that 100 hours were insufficient to establish equilibrium microstructures at these temperatures. In the sample that was treated at 1345 °C for 100 hours, a fully lamellar microstructure was produced, Figure 62. On the basis of the above, it was concluded that $1340\text{ °C} < T_{\alpha} < 1345\text{ °C}$. This is consistent with the results obtained for TiAl alloys with different forms of starting microstructures, using metallography, differential thermal analysis and dilatometry techniques [24,52,91,146]. These studies show that T_{α} ranges from 1360 to 1380 °C for Ti-48Al, Figure 63, and that each atomic % ternary addition of Cr reduces T_{α} by ~13 to 18 °C [24,91,146]. Using these observations for the present alloy (Ti-47.5Al-3Cr), a T_{α} range of 1306-1341 °C would be expected (a reduction of 39-54 °C from 1360-1380 °C). This range agrees reasonably well with the current result.

4.2.2.2 Short -Time Heat Treatments

Heat treatments were conducted at temperatures below and above T_{α} (1335, 1340, 1345, 1350, 1375 and 1400 °C) for periods from 10 to 120 minutes to determine transformation kinetics at these temperatures. The microstructures obtained are depicted

in Figures 64-67. The effects of exposure time on L_{gs} , γ_{gs} and $V\gamma_g$ are presented in Figure 61 and Table 6a. The results show that at temperatures of 1335 and 1340 °C, decomposition of the as-HIP structure was swift. After 10 minutes of heat treatment followed by air cooling, duplex microstructures with more than a two-fold increase in grain size from the as-HIP condition were obtained. When the temperature was increased to slightly above T_α at 1345 °C, the rate of lamellar grain growth increased significantly, due to the absence of γ grains that hindered α grain growth at lower temperatures. At temperatures well into the α single-phase field at 1350 °C-1400 °C, coarse-grained (~300 μm) fully lamellar structures were obtained after 10 minutes upon furnace cooling, Figure 67. The above clearly indicates that in order to obtain a fine-grained fully lamellar structure, the heat treatment temperature should be in a range of 1335°C-1340 °C, the duration should be short, and the residual γ grains need to be eliminated.

It can also be noted that the values of n for temperatures between 1320 °C and 1345 °C are higher than those at 1350 °C to 1400 °C, i.e. the rate of lamellar grain growth rate is higher at temperatures slightly below T_α . This is not surprising because when temperature approaches T_α , the amount of γ grains decreases rapidly as a result of $\gamma \rightarrow \alpha$ transformation, removing barriers for α grain growth. On the other hand, at temperature above T_α in the α single-phase field, the $\gamma \rightarrow \alpha$ transformation is complete and the α grains have to compete against each other for growth.

4.2.3 Effect of Cooling Rate

As-HIP samples were heat treated between 1300 °C and 1360 °C for two hours, followed by either air cool (AC) at a rate of 8 °C/sec, or furnace cool (FC) at a rate of 0.3 °C/sec. A slower air cooling rate of 4 °C/sec (accomplished by insulating the sample during air cooling) was also included for the 1360 °C sample. In addition, two heat treatments were conducted at 1430 °C for 20 minutes, followed by either AC or water quench. The outcome is shown in optical photographs in Figures 68-71, and in BEI in Figure 72.

At 1300 °C to 1335 °C, FC produced predominantly equiaxed γ microstructures that contained few lamellae, whereas AC produced duplex microstructures that contained both equiaxed γ grains and lamellar grains, Figures 68, 69(a,b), 72(a-d). At 1345 °C, AC and FC produced near fully lamellar microstructures that were quite different from each other, Figure 69 (c,d). In particular, in the AC sample, the volume fraction of remnant γ grains was much lower, the lamellae spacing was noticeably finer, the L_{gb} had a less interlocked morphology, and feathery γ_m was present at some L_{gb} . Also, contrast in the BEI of the AC specimen was poor, indicating that compositional differences between phases in the AC sample were much less than those in the FC sample, Figure 72 (e,f).

A fully lamellar microstructure with interlocked grain boundaries, which are beneficial for resistance against intergranular creep damage, was obtained by FC from 1360 °C, Figure 70(a). The lamellae are believed to have formed by the so-called “Type 1” reaction (i.e. $\alpha \rightarrow \alpha + \gamma_{plates} \rightarrow L(\alpha/\gamma) \rightarrow L(\alpha_2/\gamma)$) described in 2.2.4 [53, 54]. A small amount of fine equiaxed α grains was also present along the L_{gb} . They are believed to

have precipitated from α phase during slow cooling from the α single-phase field, as opposed to resulting from incomplete $\gamma \rightarrow \alpha$ transformation because the latter occurs very rapidly during heat treatment above T_α . When the cooling rate was increased to 4 °C/sec, the lamellae became finer, and feathery γ_m appeared, mostly at the L_{gb} , Figure 70(b). The L_{gb} were generally straight and smooth. Air cooling at 8 °C/sec from 1360 °C resulted in a predominantly feathery γ_m microstructure with isolated instances of lamellae, Figure 70(c). Compositional differences in both air cooled specimens were very difficult to resolve by BEI, especially in the sample that was cooled at 8 °C/sec, Figure 72 (h). The same type of feathery γ_m microstructure was also obtained after air cooling from 1400 and 1430 °C, Figure 71 (c,d). In contrast, water quenching from 1430 °C resulted in a microstructure that contains unetched featureless areas, and gray to darkly etched regions, Figure 71 (a,b). Numerous quench cracks were also produced. The unetched region was more prominent near the specimen surface where cooling rate was expected to be higher. TEM analyses on these microstructures have generally identified the light regions as α_2 phase which formed by direct ordering of the disordered high temperature α phase to α_2 during quenching, and the dark regions as the massively transformed γ phase, γ_m , which nucleates from stacking faults in the α phase [55-61,147-148]. The γ_m contains a large number of dislocations, stacking faults and anti-phase boundaries, whereas the feathery incipient γ_m consists mainly of slightly misoriented γ plates and has relatively less defects than the γ_m . There are no apparent orientation relationships and changes in composition between the parent α phase and the product phase γ_m . The presence of high density

defects in the γ_m structure indicates that it may be amenable to recrystallization by annealing.

4.2.4 Section Summary

1. The alpha transus temperature, T_α , was determined to be 1340-1345 °C.
2. Short-time heat treatments show that the $\gamma \rightarrow \alpha$ reaction is rapid not only at temperatures above T_α , but also slightly below T_α at 1335-1340 °C. The near- γ as-HIP microstructure was transformed to the fully lamellar or the duplex structure within ten minutes.
3. Heat treatments at temperatures slightly below T_α can reduce the amount of γ grains drastically and limit the lamellar grain size in near fully lamellar structures, provided the time is short and air cooling is used. Increasing the heat treatment time does not eliminate the untransformed γ grains, which tend to spheroidize and persist.
4. A cooling rate of ~ 8 °C/sec is required for the formation of unidirectional lamellae for heat treatments in the $\alpha + \gamma$ two-phase field, whereas slower cooling (furnace cooling at 0.3 °C/sec) is required for treatments in the α single-phase field.
5. Increasing the cooling rate reduces the interlocked morphology of the lamellar grain boundaries and lamellae spacing. Upon rapid cooling (water quench), the formation of lamellae is suppressed and a diffusionless massively transformed γ structure that contains high density of crystallographic defects is obtained.

From these observations, it was thought that one possible way to produce fine-grained lamellar structures from as-HIP microstructure is to conduct heat treatments at

temperatures slightly below T_α at 1335 °C-1340 °C to nucleate α phase and reduce the amount of γ grains, followed by air cool to produce a fine-grained near lamellar structure with some untransformed γ grains. The heat treatment time should be kept short (e.g. 10 minutes) to minimize α grain growth. If this procedure is repeated, more of the remnant γ grains will be transformed to α and then converted to lamellar grains.

The coarse-grained fully transformed lamellar microstructure produced by slow cooling from the α single-phase field is known to be quite stable and is unlikely to be refined by heat treatment at temperatures below T_α . This was indeed observed during exploratory tests. However, the fully transformed structure may be converted to γ_m structure first, and then subjected to the cyclic heat treatment procedure described above. This possibility was also explored and the results are presented in the following sections.

4.3 Microstructures after Cyclic Heat Treatments

4.3.1 From As-HIP Microstructure

Three cyclic heat treatment schemes were considered: 1) 12 cycles at 1335 °C for 10 minutes, followed by air cool at a rate of 8 °C/sec; 2) 6 cycles at 1340 °C/10 min./AC; 3) 12 cycles at 1330 °C/10 min./AC. The evolution of microstructures during schemes 1) and 2), and the final microstructure for scheme 3) are depicted in Figures 73 to 76, respectively. Changes in $V\gamma_g$ and L_{gs} with respect to number of cycles at 1335 °C and 1340 °C are presented in Figure 77 and Table 7. The most dramatic changes occurred during the first three cycles, especially at 1340 °C. After 12 cycles at 1335 °C and 6 cycles at 1340 °C, uniform fine-grained fully lamellar microstructures with interlocked

grain boundaries and virtually no γ grains were obtained. The lamellar grain size after treatment was 30-50 μm , 50-70 μm , and 100-150 μm for 1330 °C, 1335 °C and 1340°C, respectively. Although the finest lamellar grain size was obtained after 12 cycles at 1330 °C, approximately 10 % of γ grains still remained. It is possible that these γ grains can be further reduced by additional cycling, but the rate of reduction is expected to be very slow.

TEM examination of samples heat treated for 12 cycles at 1335 °C/10 min/AC indicates that the lamellae consist of coarse γ plates as thick as $\sim 2.5 \mu\text{m}$, interspersed with bundles of much thinner γ and α_2 plates with average plate thickness of $\sim 0.1 \mu\text{m}$. An example of the bundled arrangement is shown in Figure 78. The electron diffraction patterns (Figure 78(c)) of the coarse γ plates (γ_1 , γ_2 , and γ_3) within a lamellar grain show that they have the same crystallographic orientations, whereas the thin γ plates in the bundles are twin related. The implications of these features with respect to the formation sequence of the FGFL structure are discussed in Chapter 5.

4.3.2 Effects of Heating and Cooling Rates

A number of cyclic heat treatments with different heating and cooling rates were conducted to investigate their effects on microstructure and the formation sequence of the fine-grained lamellar microstructure. The schedules are schematically shown in Figure 79. The “reference” (RF) cycle is the baseline for comparison, it consists of heating and cooling rates of $\sim 6 \text{ }^\circ\text{C}/\text{sec}$ and $8 \text{ }^\circ\text{C}/\text{sec}$, respectively, between 1125-1370 °C. The “slow heat” (SH) cycle has a heating rate of $0.06 \text{ }^\circ\text{C}/\text{sec}$ and the slow cool (SC) cycle has a

cooling rate of 0.3 °C/sec. Two hybrid schemes, one consisting of 11 RF cycles plus 1 SC cycle, and the other including 11 RF cycles followed by 1 rapid cool (RC) cycle, were also considered to investigate the effect of final cycle cooling rate on microstructure.

The microstructures obtained are depicted in optical and BEI photos in Figure 80. In the SH sample, Figure 80 (c,d), the average γ grain size is slightly coarser ($\sim 20 \mu\text{m}$) than the RF sample ($\sim 15 \mu\text{m}$), Figure 80 (a,b). This increase may be due to the longer time spent (~ 2.5 hours per cycle) in the $\alpha+\gamma$ two-phase field during slow heating. However, this did not affect the lamellar grain size because the time spent near the maximum temperature where prior α grain growth is most rapid, was not significantly longer than in the other schemes. In contrast, cooling rate has a dramatic effect on the microstructure. Reducing the cooling rate to 0.3°C/sec from 8°C/sec produced a near- γ structure that contains approximately 10 % volume fraction of lamellar grains with extremely widely spaced lamellae, Figure 80(e,f). Even when the cooling rate was reduced in the last cycle only, the lamellae density was considerably lower than in the RF condition, Figure 80(g,h). On the other hand, increasing the cooling rate in the last cycle, as in the 11RF+1RC scheme, significantly refined the average γ and α_2 plate thickness in the bundles to $\sim 0.06 \mu\text{m}$, Figure 80(i-k), compared with $0.1 \mu\text{m}$ for the RF condition, Figure 78(b).

4.3.3 From γ_m Microstructure

As-HIP samples were heat treated in the α single-phase field at 1430 °C for 20 minutes, followed by either air cool or water quench to produce massively transformed

structures. These structures were then cyclically heat treated at 1335 °C to produce fine-grained lamellar microstructures. The microstructures before and after cyclic heat treatment are compared in Figure 81, which shows that the AC sample is predominated by the feathery incipient form of γ_m with remnants of lamellar structures, whereas the WQ sample is predominantly γ_m . It can be observed that although fine-grained fully lamellar microstructures were obtained in both cases, the WQ sample had a more uniform and finer grain size (100-150 μm) than the AC sample (200-300 μm), Figure 81(b,d). The reason is that during WQ, a high density of internal defects (anti-phase domains, dislocations and stacking faults) was created in the γ_m . These defects increased the number of nucleation sites for α phase particles during subsequent heat treatment [147-148] in the γ_m . Conversely, AC produced less defects, resulting in less nucleation sites and easier α grain growth during subsequent heat treatment. It is also noticed that many of the lamellae in the AC sample have a wavy appearance. The reason for this morphology and its effect on mechanical property are not known.

To explore the possibility of producing fine-grained fully lamellar structure from γ_m without quench cracks, a specimen was heat treated at 1370 °C for 1 hour followed by WQ. Microcracks were not detected after quenching. The sample was then cyclically heat treated at 1335 °C for 6 cycles. The resulting microstructure was a non-uniform combination of coarse lamellar grains ~200 μm in size and colonies of lamellae that have a morphology reminiscent of the feathery γ_m , Figure 82. Close examination of these colonies in BEI shows that each colony appears to contain groups of equiaxed lamellar grains with plates that are aligned somewhat differently with those of their neighbors,

Figure 82(c). The formation mechanism of these microstructures is not clear, but the above clearly indicates that γ_m can be used as a starting microstructure to develop fine-grained lamellar structures.

4.3.4 Section Summary

1. Fine-grained lamellar structures can be obtained from both as-HIP and massively transformed initial microstructures by cyclic heat treatments at temperature slightly below T_α . A range of lamellar grain sizes can be obtained by adjusting the heat treatment temperature and number of cycles.
2. After 12 cycles at 1335 °C/10 min./AC and 6 cycles at 1340 °C/10 min./AC, the amount of γ grains was practically eliminated and fine-grained fully lamellar microstructures with grain size in the ranges of 50-70 μm and 100-150 μm , respectively, were obtained.
3. Heat treating at 1330 °C for 12 cycles further reduced the lamellar grain size to 30-50 μm , but the volume fraction of residual equiaxed γ grains was ~10%.
4. Cooling rate has a greater effect on lamellar structure formation than heating rate. During cyclic heat treatments, changing the cooling rate in the last cycle substantially altered the lamellar structure (lamellar thickness and spacing, γ phase volume fraction) produced by previous cycles. A higher cooling rate reduced lamellar spacing and a lower cooling rate increased γ phase volume fraction.

On the basis of the above, two cyclic heat treatment schemes (12 cyc./1335°C/10 min./AC and 6 cyc./1340°C/10 min./AC) were chosen to produce fine-grained fully

lamellar structures from as-HIP material for mechanical property evaluation. The room temperature tensile and elevated temperature creep properties of these microstructures were compared with those of the duplex and fully lamellar microstructures. Additional microstructural conditions were also considered for creep tests, details of which are given in 4.4.2.

4.4 Room Temperature Tensile Test Results

Four microstructural conditions were considered for room temperature tensile tests: 1) duplex (DPLX) structure with a grain size of 10-35 μm ; 2) fine-grained fully lamellar structures with grain sizes of 100-150 μm (FGFL1); 3) 50-70 μm (FGFL2); 4) coarse-grained fully lamellar (FL) with a grain size of ~300-600 μm . The heat treatment schemes for tensile specimens are summarized in Table 8.

Figure 83(a) depicts the room temperature tensile stress-strain curves for the four microstructures tested. The values for 0.2% yield stress, ultimate tensile stress, total and plastic elongation are summarized in Table 8. The data obtained fall in the same range as those recently reported for forged and heat treated fine-grained lamellar materials with grain sizes ranging from 50 to 100 μm . Figures 22-23 [17, 18]. It can be observed that the tensile properties of the FGFL microstructures are approximately 10% lower than those of DPLX structure. However, their yield and tensile strengths are 10-30 % higher than the FL structure, accompanied by a three-fold increase in elongation. A Hall-Petch plot of 0.2% yield stress (YS, MPa) versus grain size ($d^{-1/2}$, $\text{m}^{-1/2}$) is presented in Figure 83(b), which shows that they can be correlated by the relationship:

$$YS = \sigma_0 + k_y d^{-0.5} = 363 + 0.6 d^{-0.5} \quad (\text{Eq. 3})$$

where k_y is the slope of the trend line. The value for k_y of 0.6 is somewhat lower than those reported in Figure 24 [11, 17] for near- γ and duplex microstructures.

Examination of the fracture surfaces in the SEM showed that failure did not appear to have initiated from any particular microstructural feature. The typical appearance of the fracture surfaces are shown in Figure 84. In the FL specimen and the FGFL1 specimen (100-150 μm grain size), the fracture mode consisted of translamellar fracture and interlamellar delamination, Figure 84(a,b). A similar fracture surface morphology was found in the FGFL2 (50-70 μm grain size) and duplex specimens, although on a much smaller scale because of their fine grain size, Figure 84 (c,d). Higher magnifications of translamellar fracture regions show that the lamellae fractured in a brittle mode with numerous transverse cracks, Figure 84(e). TEM examination of deformation substructures revealed that in the FL microstructure, deformation was accommodated by dislocations and mechanical twins. The dislocations were distributed rather non-uniformly in the lamellae, Figure 85(a). In the second mode of deformation, mechanical twins traversed across the lamellae either directly or via dislocation movements, Figure 85(b). In the FGFL microstructures, the same deformation substructures were also observed, but they were distributed in a much more uniform fashion, Figure 85(c,d).

4.5 Creep Test Results

The creep properties of six microstructures were evaluated. They include the four conditions investigated above (i.e. DPLX, FGFL1, FGFL2, FL), a FGFL3 condition (see Figure 76) produced by 12 cycles at 1330 °C/10 min./AC, and a dual-phase equiaxed (DPEQX) microstructure (Figures 68(c) and 72(a)) obtained by the same heat treatment as the DPLX, except by furnace cool. These two microstructures were included to show the qualitative effects of γ grain volume fraction on creep resistance. The results are presented in Figure 86 and summarized in Table 9. The creep curves for DPLX and FL structures are quite comparable with those shown in Figure 34 for a similar P/M material tested under slightly different conditions. The minimum strain rate as a function of grain size is plotted in Figure 87. This figure shows that the minimum creep rate decreased rapidly when the grain size was increased from ~35 μm to ~150 μm . Further increase in grain size to ~450 μm did not decrease the minimum creep rate significantly, suggesting that within this range of grain sizes, creep deformation may not be controlled by grain boundary related mechanisms. As expected, the coarse-grained fully lamellar microstructure had the longest creep life, the lowest minimum creep rate, the smallest tertiary creep region, and the lowest rupture elongation, whereas the reverse is true for the duplex and dual-phase equiaxed microstructures. It can be noted that the duplex specimens had longer rupture life and slightly lower minimum creep strain than the dual-phase equiaxed specimens, indicating the presence of lamellar grains is beneficial for creep resistance. The fine-grained fully lamellar structures exhibited creep properties between these extremes, with larger grain size resulting in better creep resistance. For

example, the minimum creep rate of the FGFL1 microstructure is ~50% higher than those of FL specimens but an order of magnitude lower than those of DPLX specimens.

Figures 88-90 show the longitudinal sections of duplex, fully lamellar and fine-grained fully lamellar specimens respectively near the fracture surface after creep test. In the DPLX and FGFL specimens, severe necking occurred. In the DPLX specimens, extensive secondary cracking in regions near the fracture surface was detected, Figure 88(a). Also, the initially equiaxed γ grains were heavily distorted and elongated in the direction of the applied load, and the presence of well defined lamellar grains was rare, Figure 88(b). TEM examination of the deformed duplex microstructure revealed a high density of dislocations and the formation of subgrain boundaries, Figure 91(a,b). Electron diffraction analysis showed the dislocations were mainly $1/2\langle 110 \rangle$ unit dislocations. Dislocation free subgrains were also observed, indicating dynamic recrystallization had occurred.

In the FL condition, the microstructure underwent relatively little distortion, even in the grains that are located on the fracture surface, Figure 89, which shows some bowing and delamination of the lamellae. Intergranular cracks were present and in some cases they appeared to be associated with coarsen lamellae or γ grains at the grain boundaries. There is some evidence that the grains with lamellae oriented at angles between $\sim 30\text{-}60^\circ$ with the loading direction (i.e. the “soft” mode of orientation reviewed in 2.3.2.3) displaying less distortion. TEM examination of thin foils taken from FL specimens showed that deformation was accommodated by dislocation activity and

twinning, Figure 91(c). However, these features were not uniformly distributed in the specimen, as many of the fine lamellae were relatively free of dislocations, Figure 91(d).

In the FGFL specimens, extensive secondary cracks were found at the lamellae grain boundaries, and the lamellae in most of the grains were heavily distorted and appeared to realign with the loading axis, Figure 90(a). In contrast, the grains with lamellae oriented in the soft mode ($\sim 30\text{-}60^\circ$ from the loading direction) displayed far less bowing, Figure 90(b). In the grains that contained lamellae that were parallel to the loading axis (i.e. in the “hard” mode orientation), shear bands sometimes developed. Under higher magnifications in the optical microscope, it appeared that the lamellae in the shear bands had disintegrated and spheriodized. The deformation substructures in the FGFL specimens are depicted in Figure 92, which shows copious amount of dislocations uniformly distributed in the coarse γ lamellae, within which subgrain boundaries were formed, Figure 92(b). In contrast, dislocations were scarce in the fine bundles of γ/α_2 lamellae and appeared to stop at coarse γ /bundle interfaces.

CHAPTER 5

DISCUSSIONS

5.1 Grain Growth and Transformation Kinetics

Figures 64-66 show that a 10-minute exposure at temperatures of 1335, 1340 and 1345 °C completely transformed the as-HIP near- γ equiaxed structure to predominantly lamellar microstructures, indicating that the $\gamma \rightarrow \alpha$ reaction is initially fast at these temperatures. As the heat treatment time was increased, the volume fraction of γ grains continued to decrease rapidly within the first 60 minutes and tapered off to a slower rate thereafter. Figure 61(a,b) and Table 6(b) show that increase in the lamellar grain size with heat treatment time can be well correlated by a power function in the form of $L_{gs} = kt^n$, indicating that growth of the α grains is controlled by diffusion related mechanisms.

After 100 hours of exposure at 1335 to 1345 °C the boundaries of the lamellar grains have become less interlocked and the lamellae are noticeably more refined. When these microstructures are examined in BEI, very little contrast/compositional difference is observed, indicating the microstructure has been homogenized. After 100 hours, some γ grains still persist; it is expected that as long as the heat treatment temperature is below T_{α} , they will not be eliminated by increasing isothermal heat treatment time. The shape of the γ grains has become less polygonal and more spherical, Figures 59-60. This morphological change is likely associated with reduced free energy under near equilibrium condition. It is interesting to note that the spheroidized γ grains are located

not only at the lamellar grain boundaries but also within the lamellar grains, which shows that the γ grains do not always impede α grain growth during long-time heat treatment.

5.2 Formation of Lamellar Microstructures

5.2.1 From as-HIP near- γ structure during cyclic heat treatment

The microstructure of samples after cyclic heat treatment is characterized by predominantly well-defined lamellar grains that consist of lamellae which appear to be coarse and widely spaced, Figures 73-76. TEM results show that the lamellae typically consist of coarse γ plates with identical crystallographic orientation, and bundles of thin γ and α_2 plates, and that the thin γ plates in the bundles have twin relationships, Figure 78. These features are different from those in the conventional fully lamellar structure which only contains α_2 and twin related γ plates (i.e. Type 1 structure as described in 2.2.4). The lamellar structures in the cyclically heat treated samples are believed to have developed as follows.

When the as-HIP near- γ microstructure is heated to slightly below T_α during the first cycle, the volume fraction of α phase increases because α is the dominant phase in this region of the $\alpha+\gamma$ two-phase field, Figure 4. This increase is achieved by:

- a) α phase precipitating out of the γ matrix of the original γ grains (as described in Type 3 lamellae formation sequence in 2.2.4) and developing into thick plates at the expense of the γ matrix;

- b) α_2 particles that are present at the γ grain boundaries of the near- γ as-HIP microstructure (Figure 54) disordering and transforming into α phase particles that coalesce and grow into α grains;
- c) the small amount of β_2 phase present in the as-HIP structure transforming into α in the $\alpha+\gamma$ two-phase field since α is the prevalent phase in this part of the phase diagram.

However, because of the short exposure time, the $\gamma \rightarrow \alpha$ transformation does not take place in some of the γ grains. The above events lead to a high temperature microstructure that consists of:

1. α grains that contain α plates and γ plates, with the latter having identical crystallographic orientation in each grain because they are from the same prior γ matrix;
2. α grains that are developed from α_2 particles in the as-HIP structure and therefore do not contain γ plates;
3. α grains as a result of coalescence of 1 and 2;
4. residual γ grains that have no or a very small amount of α plates.

Upon cooling, the volume fraction of γ phase has to increase and therefore new γ plates nucleate from α phase. In the grains that contain prior α plates and prior γ plates, γ plates nucleate and grow in the prior α plates, which subsequently transform into bundles of thin α_2 and $\gamma/\gamma_{\text{twin}}$ plates (i.e. as described in section 2.2.4 for Type 1 lamellae: $\alpha \rightarrow \alpha + \gamma_{\text{plates}} \rightarrow L(\alpha/\gamma) \rightarrow L(\alpha_2/\gamma)$). These bundles are separated by thick prior γ plates that have the same crystallographic orientation, resulting in a mixture of Type 1 and Type 3

lamellae. In the prior α grains that do not contain prior γ plates, only Type 1 lamellae are expected to develop. With regard to the residual γ grains, the ones that contain no prior α plates change little upon cooling, whereas those that contain a few prior α plates may develop lamellae with appearances similar to those shown in Figure 74(d,e). Finally, upon cooling, β_2 phase precipitate out of α and congregate predominantly at the grain boundaries as particles that are finer than their as-HIP predecessors.

During subsequent cycles, the lamellar grains that were developed in the first cycle simply convert to α grains and grow in size at the expense of smaller adjacent grains upon heating, and transform back to lamellar structure upon cooling. The increase in grain size is small over the entire period of 12 cycles because the dwell time per cycle is short. With regard to the γ grains that have not been transformed, they become potential nucleation sites for new α plates in subsequent cycles. This process is repeated as cycling continues until most of the residual γ grains are transformed into lamellar colonies.

The above pertains to lamellae formation during cyclic heat treatment at intermediate cooling rates (air cool at 8°C/sec). However, if the cooling rate employed is slow, such as that in the SC cycles (i.e. 12 cycles at 1335 °C/10 min/ furnace cool at 0.3 °C/sec cycle), a majority of the α phase transforms back to equiaxed γ , resulting in a near- γ microstructure with a small volume fraction of lamellar grains that contain widely spaced lamellae, as shown in Figures 80e and 80f.

5.3 Effect of Cooling Rate

The results in Figures 68-72 clearly show the formation of lamellar structure is contingent upon cooling rate. When static heat treatments were conducted at 1320 °C and 1335 °C in the $\alpha+\gamma$ two-phase field, air cooling produced duplex structures that contain both lamellar grains and equiaxed γ grains, whereas furnace cooling produced a near- γ microstructure with negligible amount of lamellae. On the other hand, when heat treatments were conducted in the α single-phase field, furnace cooling resulted in fully lamellar structures, whereas air cooling and water quench led to γ_m microstructures. The relationship between cooling rate and prevailing transformation processes is explained below with the assistance of a schematic continuous cooling transformation (CCT) diagram shown in Figure 93 [61,62,64]. The figure shows the starting curves for the formation of γ , lamellar, and massive γ structures overlap. Therefore, depending on the cooling rate, a combination of these structures can be produced.

5.3.1 Heat treatments near T_α in the $\alpha+\gamma$ two-phase field

In the region close to T_α in the $\alpha+\gamma$ two-phase field, α is the dominant phase, Figure 4. When the as-HIP near- γ structure is heated to this region, α phase precipitates out of the γ matrix and the α_2 phase originally present at the γ grain boundaries disorders and coarsens. These α particles subsequently grow into α grains, resulting in a high temperature microstructure that consists of predominantly α grains and residual γ grains. Upon cooling to room temperature under equilibrium condition, the phase diagram indicates that γ is the dominant phase. As a result, when the cooling rate is low, e.g. along

Path 1 in Figure 93, as in the cases of the 1320 °C and 1335 °C furnace cooled samples, γ plates nucleate from α grains to form lamellar structure as the cooling curve intersects the L_s line. However, another competing process, represented by the γ_s line in Figure 93, also begins. It involves coarsening and agglomerating of the γ particles by diffusional growth to form equiaxed γ grains. Because of the slow cool, the second process dominates, resulting in near- γ microstructures that contain very few lamellae, as shown in Figure 72(a,c). In contrast, if the cooling rate is increased to Path 2 for example, as in the cases of the air cooled samples, γ plates nucleate from α grains at a higher rate [62], but there is little time for the plates to coarsen by diffusion. Consequently, much of the α phase is retained and transforms to α_2 upon further cooling, resulting in a mixture of γ/α_2 lamellar grains and equiaxed γ grains as shown in Figure 72(b,d).

5.3.2 Heat treatments above T_α in the α single-phase field

The same processes compete with each other during cooling from the α single-phase field. When the cooling rate is low, for example along Path 3 in Figure 93 for the specimen that was furnace cooled at 0.3 °C/sec from 1360 °C, γ phase begins to nucleate from α stacking faults when the cooling curve intersects the lamellar start curve, L_s . The γ phase develop into plates, and grow coherently into the matrix of prior α grains, and incoherently into the adjacent grains, giving rise to the high temperature lamellar structure $L(\alpha/\gamma)$ with interlocking grain boundaries. Upon further cooling, α transforms to α_2 , resulting in the fully lamellar structure shown in Figure 70(a). It is noted that if the cooling rate is higher, the $\alpha \rightarrow \alpha_2$ ordering reaction can precede γ precipitation.

If the cooling rate is increased by air cool along Path 4, the formation of lamellae is interrupted by the massive diffusionless transformation of $\alpha \rightarrow \gamma$, resulting in a mixture of lamellar and feathery γ_m structures, as observed in Figure 70 (b,c). Under these conditions, the lamellar grain boundaries lose their interlocking morphology, presumably due to insufficient time for incoherent growth of the lamellae into neighbouring grains. This is an important consideration in microstructural design because interlocking grain boundaries are beneficial for creep resistance.

If the cooling rate is rapid, such as that experienced by the sample that was water quenched from 1430 °C, the cooling curve traces along Path 5 and intersects only the γ_{ms} start line. Under these conditions, the precipitation of γ particles from the α matrix (and therefore the formation of lamellae), is suppressed and the $\alpha \rightarrow \gamma$ diffusionless transformation dominates, resulting in the microstructure shown in Figure 71(a,b).

5.4 Tensile Ductility of the FGFL Microstructure

The superior room temperature tensile properties of the fine-grained fully lamellar microstructures compared with their coarse-grained counterpart is due to a grain size effect. As described in 2.3.2.3 and demonstrated by the results in Figures 26 and 27, the lamellar structure can undergo a hard mode (high strength, low ductility) or a soft mode (low strength, high ductility) of deformation, depending on its orientation with respect to the load axis. The soft mode occurs when the angle between the lamellae and the load axis is ~30-60°, whereas the hard mode occurs when the loading axis is perpendicular or parallel to the lamellae. These modes of deformation create non-uniform deformation in adjacent grains

with different lamellae orientations and strain incompatibility between the grains. In the coarse-grained fully lamellar microstructure, these effects are more pronounced because for a given volume of material, there are fewer grains in the soft orientations to accommodate the shear deformation, resulting in large strain incompatibility between grains. The reverse is true for the fine-grained fully lamellar microstructures. As a result, the fine-grained specimens deform much more uniformly, Figure 85, and are able to accommodate larger deformation before failure occurs. Figure 83(b) shows the grain size and 0.2% yield strength of the different microstructures can be described by the Hall-Petch relationship in Equation 4.1, indicating that the room temperature yield stress is predominated by a grain size effect other than by a microstructural effect

5.5 Creep Resistance of the FGFL Microstructure

The results showed the FGFL structures have much better creep resistance than the duplex structure. This is due to the absence of γ grains as well as increased lamellar grain size in the FGFL structures. As shown by the creep curves for the dual-phase equiaxed and the duplex microstructures in Figure 86(a) and the deformation structure in the duplex specimens, Figure 88, the γ grains have little resistance to creep deformation and the presence of lamellar grains are beneficial, even when the lamellar grain size was in the range of $\sim 35 \mu\text{m}$. As a result, when the volume fraction of γ grains was reduced to less than 10% and the lamellar grain size was increased slightly to $\sim 50 \mu\text{m}$, as in the fine-grained lamellar structure FGFL3, minimum creep rate was reduced by half and the rupture life was almost doubled. An order-of-magnitude reduction in minimum creep rate and a

five-fold increase in rupture life was achieved by eliminating the γ grains and increasing the lamellar grain size to $\sim 150 \mu\text{m}$. However, increasing the grain size to $\sim 450 \mu\text{m}$ did not reduce the minimum creep rate significantly, Figure 87, which suggests that creep deformation in this range of grain sizes was not dominated by grain boundary controlled mechanisms at the stress level and test temperature considered. This is consistent with the observations reported in [116,117] for other P/M and wrought alloys, Figure 37, as well as the microstructural evidence presented in Figures 91-92. The latter show a high level of dislocation activity in the coarse γ lamellae. In contrast, the fine γ/α_2 lamellae bundles contained little dislocations, and the interfaces of the bundles with the coarse γ lamellae appeared to serve as barriers to dislocation movement. Therefore, it is possible that the minimum creep rate of the FGFL structures can be further reduced by increasing the number of these bundles and interfaces. This may be accomplished by increasing the cooling rate.

Compared with the FL structure, the FGFL structures underwent extensive tertiary creep, Figure 86(a). This can be attributed to their fine grain size which accommodates deformation in a more uniform fashion, Figure 90. As a result, when secondary cracks develop at the grain boundaries during tertiary creep, they are subjected to a lower stress intensity factor and therefore propagate slowly or become arrested by other boundaries before they reach critical crack size. In contrast, deformation in the fully lamellar microstructure is non-uniform, Figure 86, due to its large grain size which amplifies strain incompatibility among adjacent grains having hard/soft deformation modes. Consequently, creep cracks develop preferentially at a few locations of strain incompatibility and grow

rapidly to reach critical size before they are impeded by obstacles, resulting in a minimal tertiary regime. It is interesting to note that the lamellae in soft oriented grains underwent much less distortion than others. This may be explained by results from previous studies that showed deformation in soft orientations is accommodated by shear in the γ lamellae on $\{111\}$ slip planes that are parallel to the lamellar boundaries [98-104].

CHAPTER 6

GENERAL DISCUSSION

The primary objective of this thesis is to develop a novel heat treatment procedure to produce fine-grained lamellar microstructures for improved mechanical property balance in TiAl alloys, using a P/M material as the starting material. The results show that this can be achieved by cyclic heat treatment at temperatures slightly below the T_α temperature in the $\alpha+\gamma$ two-phase field using as-HIP near- γ material as the initial microstructure. The technique exploits the rapid $\gamma \rightarrow \alpha$ transformation that occurs in this temperature range to promote the transformation of original γ grains to α grains, and employs a short exposure time to limit the growth of transformed α grains. Using this approach, FGFL microstructures with grain size as small as 50 μm can be obtained. This grain size is finer than those obtained by other methods reviewed in 2.2.4.. More importantly, a range of FGFL microstructures with different combinations of room temperature tensile and elevated temperature creep properties can be designed by adjusting the cyclic heat treatment temperature and the number of cycles. Because the technique is centred on the $\gamma \leftrightarrow \alpha$ transformation kinetics, alloy modifications that affect the α -transus temperature would play an important role in establishing the cyclic heat treatment parameters, and ultimately the characteristics of the fine-grained lamellar structure produced. It is plausible that large additions of T_α depressing elements (e.g. Mn, Cr, V) would be beneficial because they reduce the $\gamma \leftrightarrow \alpha$ transformation kinetics and therefore the growth of α grains during cyclic heat treatment, leading to a more refined

microstructure. When compared with the standard fine-grained duplex and the coarse-grained fully lamellar microstructures, the FGFL microstructures have much better mechanical property balance. On the basis of the results in other studies reviewed in Chapter 2, it is expected that the FGFL structures will also provide similar improvements in other mechanical properties, including fracture toughness and fatigue.

The FGFL microstructures obtained in the present study have not been optimized. It is likely that their mechanical properties can be further improved, for example, by refining the width of the coarse γ lamellae and decreasing the lamellar spacing; both are shown to have played an important role in dislocation movements in the microstructure. Figures 85, 91, 92. In this regard, the lamellae spacing can be refined by increasing the cooling rate at the last cycle, as demonstrated in Figure 80. More work is required in this area to elucidate the effects of different microstructural parameters in the FGFL structures on mechanical properties.

The results presented in Figure 81 show that the cyclic heat treatment can also transform massive γ structure to a FGFL microstructure, which suggests that in principle, the technique can be used to refine other forms of materials with different compositions. For example, the initial coarse-grained lamellar structure in as-cast materials can be first converted to γ_m , which provides the driving force and nucleation sites for recrystallization during subsequent cyclic heat treatment. However, thermal cracking can occur during rapid cool, as in the case for the water quenched specimen in Figure 81(a). Although cracking in the sample was avoided by reducing the cooling rate to air cool, but the formation of γ_m was incomplete, and the driving force was insufficient to produce

uniform fine-grained lamellar microstructures during subsequent cyclic heat treatment, as shown in Figure 81(d). This dilemma may be circumvented because there is evidence that the cooling rate required to produce γ_m depends on alloy composition [116,117,147, 148], i.e. the location of the γ_{ms} start curve in Figure 93 is influenced by composition. It has been shown that air cooling a P/M binary alloy Ti-48Al from 1400 °C in the α single-phase region produced a small amount of massive γ at the grain boundaries [116,117], whereas with ternary addition of Cr, as in the current material, a significant amount of γ_m developed during air cooling from 1360 °C, Figure 70(c). These effects are consistent with those reported recently in [147,148], which show the formation of γ_m depends on alloy composition, the initial microstructure, as well as the temperature from which quenching is applied.

The cyclic heat treatments in this study have been conducted manually in a conventional radiation furnace, and the specimens need to be encapsulated to avoid oxidation at high temperature, both procedures are unsuitable for practical applications. The technique can be simplified by using a vacuum furnace, but it may be limited by the heating rate and the time required for evacuation of the particular furnace. A possible alternative is induction heating, which will provide much better flexibility in heating and cooling rates. However, some form of protection for the specimen/component against oxidation is required. In this regard, little has been reported on the protection of TiAl alloys against oxidation and corrosion damage. This is clearly a subject of practical importance as the demand for high temperature performance continues.

CHAPTER 7

SUMMARY AND CONCLUSIONS

1. A literature review on the effects of microstructure on the mechanical properties of near- γ TiAl alloys shows that a “fine-grained fully lamellar” (FGFL) microstructure, produced in cast and wrought alloys by elaborate thermomechanical processing and compositional modifications, improves the mechanical property balance in γ -TiAl alloys. In this thesis, a novel procedure that utilizes heat treatment only, has been successfully developed to produce FGFL microstructures in a hot isostatically consolidated powder metallurgy alloy Ti-47.5Al-3Cr.
2. The technique involves heat treatment at temperatures slightly below the α -transus temperature in a cyclic manner to exploit the rapid $\gamma \rightarrow \alpha$ transformation that occurs in this temperature range, and employs a short exposure time to limit the growth of transformed α grains.
3. Using this approach, a range of FGFL microstructures with different combinations of mechanical properties could be obtained by adjusting the heat treatment temperature and the number of cycles. The FGFL structures exhibited minimum creep rate an order of magnitude lower than that of duplex microstructure, and with approximately 10% reduction in tensile properties. When compared to the fully lamellar structure, the FGFL structures displayed a three-fold increase in tensile elongation and 30 % loss in minimum creep rate.
4. The results also show that FGFL structures were produced by cyclically heat treating a massively transformed γ structure. Therefore, in principle, the procedure can be used to transform coarse-grained inhomogeneous cast materials to FGFL structures by first converting the cast structure to a massive γ structure via rapid cooling, followed by

applying the cyclic heat treatment. However, the potential problem of thermal cracking during rapid cool needs to be addressed.

5. The FGFL microstructure may be optimized further by decreasing the lamellar spacing, which is believed to be beneficial for mechanical properties including creep. This can be accomplished by increasing the cooling rate in the last cycle.
6. A series of isothermal heat treatments has been conducted to determine the α -transus temperature, T_α , the $\gamma \rightarrow \alpha$ transformation kinetics, and the effect of heating and cooling rates on the microstructure of P/M Ti-47.5Al-3Cr. Results show that $1340^\circ\text{C} < T_\alpha < 1345^\circ\text{C}$, and that the formation and morphology of the lamellae depends significantly on cooling rate. Increasing the cooling rate reduces the lamellae spacing, as well as the interlocking grain boundary morphology of the lamellar grains. Upon rapid cooling (water quench), the formation of lamellae is suppressed and a diffusionless massively transformed γ structure that contains high density of crystallographic defects is obtained. The massive γ structure was converted to FGFL microstructure by cyclic heat treatment.

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TABLE 1
COMPARISON OF THE PROPERTIES OF TITANIUM ALUMINIDES WITH
CONVENTIONAL TITANIUM ALLOYS AND SUPERALLOYS [2-4, 9].

PROPERTY	α_2 -Ti ₃ Al	γ -TiAl	Titanium Alloys	Superalloys
Structure	DO ₁₉	L1 ₀	hcp/bcc	fcc/L1 ₂
Density, g/cm ³	4.1-4.7	3.7-3.9	4.5	8.3
Room Temp.:				
Modulus, GPa	120-145	160-176	96-115	206
Yield, MPa	700-900	400-970	380-1150	800
UTS, MPa	800-1140	450-1000	480-1200	1300
Ductility, %	2-7	1-4	10-20	3-5
K _{IC} , MPa√m	15-20	10-35	50-80	> 100
Charpy, J	< 4	0.5-5	25	30
High Temp.:				
Creep limit, °C	730	900	600	1090
Oxid. limit, °C	650	815	600	1090
Ductility, %	5-12	5-12	15	10-20

TABLE 2
PARTIAL LIST OF MECHANICAL PROPERTY REQUIREMENTS
FOR AERO ENGINE COMPONENTS [71].

Components	Advanced Compressor	HP Compressor Vanes	HP Compressor Blades	Aft Compressor Casing	Exhaust (vanes, cones, ducts, mixers)
Yield strength / Ultimate tensile strength, MPa / elongation, %					
25 °C	627/723	517/586/3	517/586/3	517/586/3	n.a.
650 °C	585/675	n.a.	n.a.	n.a.	n.a.
730 °C	537/655	n.a.	n.a.	n.a.	n.a.
760 °C	n.a.	414/517/4	414/517/4	414/517/4	n.a.
815 °C	482/551	n.a.	n.a.	n.a.	n.a.
870 °C	n.a.	n.a.	n.a.	n.a.	310/445/5
980 °C	n.a.	n.a.	n.a.	n.a.	207/379/>10
High Cycle Fatigue Strength, MPa					
10 ⁹ cycles R = 0.05 <u>K_t = 2.16</u>					
25 °C	338				
650 °C	303	n.a.	n.a.	n.a.	n.a.
730 °C	289				
3x10 ⁷ cycles <u>K_t = 3</u>	n.a.	207	207	n.a.	n.a.
Low Cycle Fatigue Strength, MPa					
10 ⁷ cycles R = 0.05 <u>K_t = 1:</u>					
25 °C	648				
650 °C	599	n.a.	n.a.	n.a.	n.a.
730 °C	551				
<u>K_t = 2.16:</u>					
25 °C	455				
650 °C	386	n.a.	n.a.	n.a.	n.a.
730 °C	351				
10 ⁵ cycles <u>K_t = 1.4</u>	n.a.	207	76	n.a.	n.a.
760 °C					
10 ⁴ cycles <u>K_t = 2.0</u>					
760 °C	n.a.	n.a.	n.a.	172	n.a.
Creep Resistance					
760 °C 500 hr	n.a.	0.2% elong. at 69 MPa	0.1% elong. at 103 MPa	0.2% elong. at 138 MPa	n.a.
870 °C 25 hr	n.a.	n.a.	n.a.	n.a.	1% elong. at 172 MPa
980 °C 25 hr	n.a.	n.a.	n.a.	n.a.	1% elong. at 41 MPa
Fracture Toughness: 27.5 MPa√m at 25 °C					
Fatigue Crack Growth: set inspection period for a given initial flaw size.					
Oxidation/Corrosion Resistance: as required					

K_t = elastic stress concentration factor; R = minimum applied stress ÷ maximum applied stress; n.a. = not available.

TABLE 3
EFFECTS OF COMPOSITION AND ALLOYING ELEMENTS ON THE
PROPERTIES OF TWO-PHASE NEAR-GAMMA ALLOYS [20,21,34].

Elements	Reported Effects
Al	Strongly affects ductility by changing the microstructure. The best ductility occurs in the range of 46-50 at.%. Within this range, increasing Al content decreases fracture toughness, decreasing Al content increasing amount of α_2 phase and strength.
B	Additions of >0.5 at.% refine cast microstructure. Improves strength by dispersion or precipitation hardening. Doping decreases melt viscosity and increases castability.
C	Doping increases creep resistance but decreases ductility. Improves strength by dispersion or precipitation hardening.
Cr	Additions of 1-3 at.% increase the ductility of duplex alloys; additions >2 at.% enhance workability and superplasticity. Generally reduces oxidation resistance, but additions >8 at.% greatly improve oxidation resistance. Provides solid solution strengthening. Shifts the $(\alpha_2+\gamma)/\gamma$ to the titanium side and decreases Al content in the γ phase.
Fe	Increases fluidity but becomes more susceptible to hot cracking.
Mn	Additions of 1-2 at.% increase the ductility of duplex alloys. Reduces oxidation resistance. Provides solid solution strengthening. Shifts the $(\alpha_2+\gamma)/\gamma$ to the titanium side and decreases Al content in the γ phase.
Mo	Improves the ductility and strength of a fine grained material; also improves oxidation resistance.
N	Improves creep resistance. Provides dispersion or precipitation strengthening.
Ni	Increases fluidity.
Nb	Enhances oxidation resistance, improves creep resistance slightly, but produces dendritic segregation. Provides effective solid solution strengthening.
P	Doping increases the oxidation resistance.
Se	Minor additions increases oxidation resistance remarkably.
Si	Increases fluidity, reduces susceptibility to hot cracking, refines cast microstructure and improves oxidation resistance. Additions of 0.5-1 at.% improve creep resistance. Lowers fracture toughness.
Ta	Improves oxidation and creep resistance, but increases susceptibility to hot cracking and dendritic segregation. Provides solid solution strengthening.
Te	Minor additions increases oxidation resistance remarkably.
V	Additions of 1-3 at.% increase the ductility of duplex alloys, but generally reduce the oxidation resistance. Provides solid solution strengthening. Shifts the $(\alpha_2+\gamma)/\gamma$ to the titanium side and decreases Al content in the γ phase.
W	Greatly improves creep and oxidation resistance. Provides solid solution strengthening.
Zr	Increases creep resistance but reduces fracture toughness.

TABLE 4
HIGH CYCLE FATIGUE PROPERTIES OF TIAL ALLOYS IN AIR [43].

Composition	Form and Microstructure	Test Conditions	Temp. °C	Yield Strength MPa	Fatigue Strength★ MPa	FS/YS
Ti-48Al-1V-0.2C	forging; DP	R = -1 f = 40 Hz	25	480	475	0.98
		"	815	360	250	0.69
	casting; FL	"	25	400	325	0.81
		"	800	350	185	0.53
Ti-48Al-2Nb-2Cr	casting; DP	R = 0.1 f = 60 Hz	760	331	290	0.88
	extrusion; DP	A = 1.0 f = 30 Hz	650	404	275	0.68
		"	760	402	174	0.43
	extrusion; FL	"	650	410	275	0.67
		"	760	408	215	0.53
Ti-47Al-2Mn-2Nb-0.8TiB ₂	casting; NL	R = 0.1 f = 60 Hz	650	369	340	0.99
		"	760	344	340	0.94
Ti-45Al-2Mn-2Nb-0.8TiB ₂	casting; TMT	"	650	476	449	0.94
		"	760	415	345	0.83
Ti-46.5Al-2Cr-Nb-0.2W ("Alloy K5")	forging; DP	R = 0.1 f = 25 Hz★★	600	414	530	1.28
	forging; RFL	"	800	418	245	0.59
		"	600	407	470	1.15
		"	800	375	315	0.84
		"	870	362	250	0.70

DP =duplex; FL =fully lamellar; NL =nearly lamellar; TMT =thermomechanically treated; RFL =refined fully lamellar. R =minimum applied stress÷maximum applied stress; compressive stress being negative; A =alternating stress amplitude ÷ mean stress; f =test frequency.

★ =fatigue strength at 10⁷ cycles run-out; ★★ =tests conducted at a strain rate of 5 x 10⁻² s⁻¹ ; all others are assumed to be tested at a rate in the range of 1 x 10⁻³ s⁻¹ to 1 x 10⁻⁴ s⁻¹ .

TABLE 5
CHEMICAL COMPOSITION OF PREALLOYED POWDERS

	Al	Cr	Fe	C	N	O	H	Ti
Wt. %	33.4	4.0	.020	.014	.004	.092	.003	bal.
At. %	47.1	2.9	.013	.044	.011	.219	.113	49.6

TABLE 6
(a) EFFECTS OF ISOTHERMAL HEAT TREATMENT TIME AND TEMPERATURE ON LAMELLAR GRAIN SIZE AND VOLUME FRACTION OF EQUIAXED γ GRAINS.

Temp.	L_{gs} , $V_{\gamma g}$	Heat Treatment Time						
		10 min	30 min	60 min	2 hr	25 h	50 h	100 h
1300°C	L_{gs} , μm	--	--	--	--	--	--	--
	$V_{\gamma g}$, %	--	--	--	63	--	--	--
1320°C	L_{gs} , μm	--	--	--	30	40	60	80
	$V_{\gamma g}$, %	--	--	--	33	25	22	16
1330°C	L_{gs} , μm	--	--	--	40	60	75	100
	$V_{\gamma g}$, %	--	--	--	16	15	14	14
1335°C	L_{gs} , μm	25	30	36	50	112	130	150
	$V_{\gamma g}$, %	30	24	18	12	11 (16 hr)	10 (60 hr)	10
1340°C	L_{gs} , μm	33	39	45	70	120	134	190
	$V_{\gamma g}$, %	20	13	10	8	7	7	6
1345°C	L_{gs} , μm	94	154	185	270	--	--	500
	$V_{\gamma g}$, %	11	8	5	3	--	--	0
1350°C	L_{gs} , μm	283	--	--	392	--	--	--
	$V_{\gamma g}$, %	0	--	--	--	--	--	--
1375°C	L_{gs} , μm	306	--	--	447	--	--	--
	$V_{\gamma g}$, %	0	--	--	--	--	--	--
1400°C	L_{gs} , μm	325	406	461	492	--	--	--
	$V_{\gamma g}$, %	0	--	--	--	--	--	--

L_{gs} = lamellar grain size in μm ; $V_{\gamma g}$ = % volume of equiaxed γ grains.

All samples were air cooled at 8 °C/sec after heat treatment.

(b) LAMELLAR GRAIN SIZE AS A FUNCTION OF ISOTHERMAL HEAT TREATMENT TEMPERATURE FOR HEAT TREATMENT TIME OF 2 HOURS.

Temperature. T	$L_{gs} = kt^n$		Coef. Corr.
	k	n	r^2
1320°C	9.1	0.24	0.94
1330°C	13.3	0.22	0.94
1335°C	11.6	0.3	0.98
1340°C	16.5	0.27	0.98
1345°C	64.7	0.25	0.93
1350°C*	209	0.13	-
1375°C*	215	0.15	-
1400°C	227	0.17	0.97

L_{gs} = lamellar grain size (μm); t = heat treatment time (minutes)

* based on only two values of t.

TABLE 7
EFFECTS OF CYCLIC HEAT TREATMENT TEMPERATURE
AND CYCLES ON LAMELLAR GRAIN SIZE AND
VOLUME FRACTION OF EQUIAXED γ GRAINS.

Temp.	L_{gs} , $V_{\gamma g}$	Heat Treatment Cycles				
		1	3	6	9	12
1330°C	L_{gs} , μm	--	--	--	--	30-50
	$V_{\gamma g}$, %	--	--	--	--	10
1335°C	L_{gs} , μm	25	30	40	50	50-70
	$V_{\gamma g}$, %	30	15	5	2	1
1340°C	L_{gs} , μm	33	120	100-150	--	--
	$V_{\gamma g}$, %	20	2	0.3	--	--

L_{gs} = lamellar grain size in μm ; $V_{\gamma g}$ = % volume of equiaxed γ grains.

All samples were air cooled at 8 °C/sec after each cycle.

TABLE 8
EFFECTS OF MICROSTRUCTURE ON ROOM
TEMPERATURE TENSILE PROPERTIES.

Microstructural Condition (Heat Treatment Scheme)	Elastic Modulus GPa	0.2% Yield Stress MPa	Ultimate Tensile Stress MPa	Total/Plastic Elong. %
"DPLX" Fine-grained duplex, grain size 25 μm (1320 °C /2h/AC) + (950 °C/ 48h/ FC)	148	460	536	2.06/1.7
"FGFL1" Fine-grained fully lamellar, g.s. 100-150 μm (6 cycles /1340 °C/10 min/AC) + (950 °C/ 48h/ FC)	147	411	523	1.88/1.5
"FGFL2" Fine-grained fully lamellar, g.s. 50-70 μm (12 cycles /1335 °C/10 min/AC) + (950 °C/ 48h/ FC)	141	424	538	2.24/1.9
"FL" Coarse-grained fully lamellar, g.s. 400-500 μm (1350 °C/2h/FC) + (950 °C/ 48h/ FC)	123	372	384	0.65/0.33

TABLE 9
EFFECTS OF MICROSTRUCTURE ON CREEP RUPTURE PROPERTIES
AT 760 °C, INITIAL STRESS OF 240 MPa.

Microstructural Condition (Heat Treatment Scheme)	Rupture life, Tr hour	Rupture strain, ϵ %	Minimum strain rate, $\dot{\epsilon}_{min}$, h^{-1}
“DPEQX” Dual phase equiaxed, grain size 35 μm (1320 °C /2h/FC) + (950 °C/ 48h/ FC)	38.5 34	40.8 37	2×10^{-3} 1.8×10^{-3}
“DPLX” Duplex, grain size 35 μm (1320 °C /2h/AC) + (950 °C/ 48h/ FC)	63 58	32 32.8	1.5×10^{-3} 1.9×10^{-3}
“FGFL1” Fine-grained fully lamellar, g.s. 100-150 μm (6 cycles /1340 °C/10 min/AC) + (950 °C/ 48h/ FC)	294	19.7	0.17×10^{-3}
“FGFL2” Fine-grained fully lamellar, g.s. 50-70 μm (12 cycles /1335 °C/10 min/AC) + (950 °C/ 48h/ FC)	160 194	31.8 22	0.4×10^{-3} 0.38×10^{-3}
“FGFL3” Fine-grained fully lamellar, g.s. 30-50 μm (12 cycles /1330 °C/10 min/AC) + (950 °C/ 48h/ FC)	90 103	32.6 32.9	1.2×10^{-3} 1.1×10^{-3}
“FL” Coarse-grained fully lamellar, g.s. 300-600 μm (1350 °C/2h/FC) + (950 °C/ 48h/ FC)	473 537	9.8 11.2	0.13×10^{-3} 0.12×10^{-3}

Figure 1 Potential applications of α_2 -Ti₃Al and γ -TiAl alloys in an aero engine [1].

Figure 2 Typical processing routes for γ -TiAl alloys. IM = ingot metallurgy; PM = powder metallurgy; HIP = hot isostatic processing; HP = hot pressing; NNS = near net shape [10].

a) Near-Gamma

b) Duplex

c) Near Fully Lamellar

d) Fully Lamellar

Figure 3 Typical microstructures in γ -TiAl alloys: a) near-gamma, arrows indicate α_2 particles; b) duplex, arrows indicate γ grains; c) near fully lamellar, arrows indicate γ grains; d) fully lamellar.

Figure 4 Binary TiAl phase diagram: a) schematic representation [26]; b) enlarged central portion of a); 'C' represents typical composition range of two-phase alloys of interest; T_{α} is the alpha transus temperature line; T_e is the eutectoid temperature; T_0 to T_4 are examples for discussion in the text.

Figure 5 Schematic comparison of mechanical properties of different microstructures, after reference [10].

Figure 6 Lattice and slip planes of γ phase: a) ordered face-centred tetragonal L1₀ structure with (111) plane hatched; b) dislocations on (111) plane [19].

Figure 7 Lattice and possible slip planes of α_2 phase : a) ordered hexagonal DO19 structure; b) possible slip planes and slip vectors in the structure [19].

Figure 8 Effect of ternary additions of Mn, Cr and V on α transus temperature [20].

Figure 9 Example of a cast-duplex microstructure produced by annealing as-cast material in the $(\alpha+\gamma)$ two-phase field for 40 hours. Arrows indicate gamma grains.

Figure 10 Optical micrograph showing the microstructure of a cast + forge near-gamma alloy with residual lamellar grains (arrow 'A'), a banded mixture of γ grains and α_2 particles (arrow 'B').

Figure 11 Example of a fine-grained duplex microstructure in a powder metallurgy processed near-gamma alloy. Arrow 'A' indicates equiaxed γ grains. arrow 'B' indicates lamellar grains consisting of α_2/γ lamellae.

Figure 12 Example of a near fully lamellar microstructure in a near-gamma alloy. Arrows indicate gamma grains.

Figure 13 Example of a fully lamellar microstructure: a) optical micrograph of an as-cast near-gamma alloy; b) TEM bright field photo showing α_2 and γ laths: inset showing selected area electron diffraction pattern for both α_2 and γ laths.

Figure 14 Effects of cooling rate from the α single-phase field on microstructure: a) a Widmanstätten-like “basket-weave” structure resulting from fast air cool; b) a massively transformed microstructure resulting from water quench, arrows indicate α grain boundaries.

Figure 15 Microstructure of a cast near-gamma alloy: a) without TiB_2 addition; b) with 0.8 vol.% TiB_2 addition, arrows indicate TiB_2 particles.

Figure 16 The effects of boron additions on the grain size of a Ti-47Al binary alloy, and a Ti-46.5Al-2Cr-3Nb-0.2W multicomponent alloy ("K5") after forging + heat treatment in the α single-phase field for 1 hour [11].

Figure 17 Effects of aluminum content, in at.%, on the room temperature tensile properties of cast alloys. [5].

Figure 18 Effects of aluminum content and microstructure on the room temperature tensile properties of: a) a cast+forged+heat treated binary alloy; b) a rapidly solidified+HIP+extruded+heat treated binary alloy [89, 90].

Figure 19 The effect of Al content and ternary additions of Cr, Mn and V on the ductility of wrought γ -TiAl alloys. Alloys that contain 44% Al are fully lamellar. 46-50% Al are duplex, and 52-54% Al are single-phase gamma [20].

Figure 20 Room temperature tensile properties of cast, forged and heat treated Ti-47Al-1Cr-1V-2.5Nb with different microstructures and grain sizes. FL= fully lamellar (shaded area represents the scatter band of FL data); NL= nearly lamellar; NG=near- γ ; GS= grain size; L= lamellar grains [34].

Figure 21 The effects of microstructure on the room temperature tensile properties of a cast, forged and heat treated Ti-46.6Al-2.3V-0.9Cr alloy. Grain sizes for the microstructures are: fully lamellar = 1200 μm ; nearly lamellar = 220 μm ; near gamma = 140 μm ; fine-grain lamellar = 52 μm ; duplex = 30 μm . [17].

Figure 22 The effect of grain size on: a) the tensile ductility and b) the ultimate strength of different microstructures. FL= fully lamellar microstructure; DM= duplex microstructure; NG= near-gamma microstructure [18].

Figure 23 The effect of grain size on the tensile yield strength of forged Ti-46.6Al-2.3V-0.9Cr alloy heat treated to fully lamellar (FL), duplex (DM) and near-gamma (NG) microstructures [18].

Figure 24 Hall-Petch relations for the room temperature tensile yield stress of single-phase gamma, duplex, and fully lamellar microstructures [11].

Figure 25 Effects of test temperature on the tensile properties of various γ -TiAl alloys in different microstructural conditions. Dotted line represents the trend of brittle-ductile transition temperature [10].

Figure 26

Room temperature tensile properties of Ti-49.5Al with different lamellae orientations. ϕ is the angle between the lamellae and the load axis: a) tensile stress-strain curves; b) tensile elongation to fracture [100].

Figure 27 Room temperature tensile test result for a Ti-49.3Al bicrystal: a) schematic and b) photo showing lamellae orientations in upper and lower grains; c) stress-strain curve of the bicrystal [101].

Figure 28 The effect of microstructure and grain size on the room temperature fracture toughness of γ -TiAl alloys [18].

Figure 29 Effects of volume fractions of equiaxed γ grains and lamellar grains on the room temperature fracture toughness of Ti-48at.%Al-2V-2Mn alloy [113].

a) Ti-44at%Al (as-cast)

b) Ti-48at%Al (as-cast)

c) Ti-50at%Al (as-cast)

d) Ti-50at%Al (I.F.-A)

Figure 30

Schematic illustration of typical crack paths in γ -TiAl alloys with: a) fully lamellar structure with smooth grain boundaries; b) fully lamellar structure with serrated grain boundaries; c) duplex microstructure with a mixture of lamellar and single-phase γ grains; d) single-phase γ microstructure [88].

Figure 31 Effect of lamellae spacing, λ , on the fracture toughness of lamellar alloys with different grain sizes [105]. K_{IC} = crack initiation toughness; K_S = crack growth toughness; G1L=Ti-47Al-2.6Nb-2(Cr+V), G8L=Ti-46.8Al-2.3Nb-1.6Cr-0.9V, K5= Ti-46.5Al-2Cr-3Nb-0.2W; K7= Ti-47Al-1.5Cr-0.5Mn-0.2B.

Figure 32 Effect of grain size on the fracture toughness of γ -TiAl alloys with fully lamellar microstructure (FL); duplex microstructure (DM); and near-gamma (NG) microstructure [18].

Figure 33 Effect of microstructure on the creep behaviour of γ -TiAl alloys in duplex, single-phase and fully lamellar microstructural conditions [25].

Figure 34 Effect of lamellar grain volume fraction on the creep behaviour of P/M Ti-48Al-3Cr alloy [44].

Figure 35 Stress rupture strength of Ti-48Al-2Cr-2Nb alloy with a duplex microstructure (lamellar grain size 40 μm , γ grain size 15 μm) and a fully lamellar structure (grain size 0.5-1mm) [115].

Figure 36 Comparison of the creep rates of a fine-grain lamellar microstructure with those of coarse-grain lamellar microstructures [65].

Figure 37 Effect of lamellar grain size on the creep rate of: a) P/M Ti-48.4Al-1.9W tested at 760 °C, 276 MPa; b) Ti-48Al-2Cr-2Nb tested at 850 °C, 250 MPa. [116, 117].

Figure 38 Comparison of the fatigue lives of a forged Ti-46.5Al-2Cr-3Nb-0.2W alloy with a duplex (DP) and a refined fully lamellar (RFL) microstructure [43].

Figure 39 Effects of microstructure on the fatigue crack growth rates in various alloys: a) Ti-46.5Al-2Cr-3Nb-0.2W (K5), Ti-48Al-2Cr-2Nb, Ti-47Al-2Cr-2Nb, Ti-46Al-5Nb-1W (Alloy 7) [70]; b) Ti-47.3Al-2.3Nb-1.5Cr-0.4V at various temperatures; R = 0.1, frequency 25 Hz in air [145].

Figure 40 Fatigue crack growth behavior of Ti-47Al-0.9Cr-0.8V-2.6Nb with a fully lamellar microstructure, showing lower propagation rates when crack growth direction is perpendicular to the lamellae [142].

Figure 41 Fatigue crack growth behavior of Ti-48Al-2Cr-2Nb with a fully lamellar microstructure, showing crack arrest at a boundary (indicated by arrows) separating two grains with different lamellae orientations. Crack growth resumed when the applied stress intensity factor was increased [144].

Figure 42 Size distribution of powder material.

Figure 43 Photograph showing product form at different stages of processing: a) powder material; b) stainless steel or titanium tube with bottom end welded; c) evacuated can ready for hot isostatic processing; d) consolidated can; e) specimen compact and tantalum foil; f) specimen encapsulated in quartz tube for heat treatment, the ends of the specimen wrapped with tantalum wire to avoid contact with quartz tube; g) mechanical test specimen.

Figure 44 Photograph showing hydraulically operated crimping tool for preparing sealed evacuated can.

Figure 45 Photograph of vacuum system consisting of a roughing pump and a diffusion pump for degassing: a) roughing pump; b) diffusion pump; c) filled can being evacuated; d) vertical furnace.

Figure 46 Photograph of hermetically sealed glove chamber for filling the cans with an argon atmosphere.

Figure 47 Schematic representation of the hot isostatic press

Figure 48 Typical thermal and pressure history of a HIP cycle with furnace cool.

Figure 49 Specimen heating rates during heat treatment.

Figure 50 Specimen cooling rates during heat treatment.

Figure 52 Typical tensile test setup.

Figure 53 Constant-load creep test setup.

a

b

c

Figure 54 Microstructure after HIP consolidation at 1250 °C/200 MPa/2h: a) optical micrograph; b) SEM BEI; c) spectrum analysis on particles indicated by arrows showing particles are rich in Cr.

Figure 55a Optical photographs showing the effect of post-HIP heat treatment temperature on microstructure. Samples were heat treated at 1300, 1320 and 1330 °C for two hours, followed by air cool (AC) at 8 °C/sec.

Figure 55b Optical photographs showing the effect of post-HIP heat treatment temperature on microstructure. Samples were heat treated at 1335, 1340 and 1345 °C for two hours, followed by air cool (AC) at 8 °C/sec.

1350 $^{\circ}\text{C}/2\text{h}/\text{AC}$

1360 $^{\circ}\text{C}/2\text{h}/\text{AC}$

1400 $^{\circ}\text{C}/2\text{h}/\text{AC}$

Figure 55c Optical photographs showing the effect of post-HIP heat treatment temperature on microstructure. Samples were heat treated at 1350, 1360 and 1400 $^{\circ}\text{C}$ for two hours, followed by air cool (AC) at 8 $^{\circ}\text{C}/\text{sec}$. γ_m = massively transformed gamma phase.

Figure 56 Effect of isothermal heat treatment temperature on the volume fraction of equiaxed γ grains.

Figure 57 Effect of isothermal heat treatment duration at 1320 °C on microstructure; AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

1330 °C/2h/ AC

1330 °C/25h/AC

1330 °C/50h/ AC

1330 °C/100h/AC

Figure 58 Effect of isothermal heat treatment duration at 1330 °C on microstructure; AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

Figure 59 Effect of isothermal heat treatment duration at 1335 °C on microstructure; AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

1340 °C/2h/AC

1340 °C/25h/AC

1340 °C/50h/AC

1340 °C/100h/AC

Figure 60 Effect of isothermal heat treatment duration at 1340 °C on microstructure; AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

Figure 61 Effects of isothermal heat treatment temperature and time on: a) the volume fraction of equiaxed γ grains; b) lamellar grain size; open circles= average γ grain size.

Figure 62 Fully lamellar structure after heat treating at 1345 °C for 100 hours, followed by air cooling at 8 °C/sec.

Figure 63 Effects of ternary Cr addition and aluminum content on alpha transus temperature of binary γ -TiAl alloys; after [20, 24, 90].

Figure 64 Effect of isothermal heat treatment duration at 1335 °C on microstructure; AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

1340 °C/10 min./AC

1340 °C/30 min./AC

1340 °C/60 min./AC

1340 °C/120 min./AC

Figure 65 Effect of isothermal heat treatment duration at 1340 °C on microstructure; AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

1345 °C/10 min/AC

1345 °C/30 min/AC

1345 °C/60 min/AC

1345 °C/120 min/AC

Figure 66 Effect of isothermal heat treatment duration at 1345 °C on microstructure; AC = air cooled at 8 °C/sec. Arrow indicate examples of γ grains.

(a) 1350 °C/10 min/FC

(b) 1375 °C/10 min/FC

(c) 1400 °C/10 min/FC

Figure 67 Microstructures after heat treating at: a) 1350 °C; b) 1375 °C; c) 1400 °C for 10 minutes. FC = furnace cooled at 0.3 °C/sec.

Figure 68 Optical micrographs showing the effect of cooling rate on the microstructure of samples heat treated at 1300 °C (a, b); and 1320 °C (c, d), for two hours. FC = furnace cooled at 0.3 °C/sec; AC = air cooled at 8°C/sec.

Figure 69 Optical micrographs showing the effect of cooling rate on the microstructure of samples heat treated at 1335 °C (a, b); and 1345 °C (c, d), for two hours. FC = furnace cooled at 0.3 °C/sec; AC = air cooled at 8°C/sec.

(a) Furnace cooled at 0.3 °C/sec

(b) Air cooled at 4 °C/sec

(c) Air cooled at 8 °C/sec

Figure 70 Effect of cooling rate on the microstructure of samples after heat treating at 1360 °C for two hours. γ_m = feathery massively transformed gamma phase; L = lamellar structure.

Figure 71 Effect of cooling rate on microstructure: a) after heat treatment at 1430 °C for 20 minutes, followed by water quench (WQ); b) another area in sample a); c) same treatment as in a) but air cooled (AC); d) after heat treatment at 1400 °C for 20 minutes, followed by AC.

Figure 72 a) SEM BEI of specimens heat treated at: (a, b) 1320 °C, and (c, d) 1335 °C for two hours, followed by either furnace cool (FC) at 0.3 °C/sec (a, c) or air cool (AC) at 8 °C/sec (b, d). Arrows indicate examples of γ grains.

Figure 72

b) SEM BEI of specimens heat treated at: (e, f) 1345 °C and (g, h) 1360°C for two hours followed by either furnace cool (FC) at 0.3 °C/sec (e, g) or air cool (AC) at 8 °C/sec (f, h). Arrows indicate examples of γ grains. γ_m = incipient form of massively transformed gamma phase.

Figure 73 Microstructures after cyclic heat treatment at 1335 °C: a) 3 cycles; b) 6 cycles; c) 9 cycles; d) 12 cycles. AC = air cooled at 8 °C/sec. Arrows and squares indicate examples of γ grains.

Figure 74 BEI of microstructures after cyclic heat treatment at 1335 °C/10 min./AC: a) 0 cycle i.e. as-HIP condition; b) 1 cycle; c) 3 cycles; d) 6 cycles; e) 9 cycles; f) 12 cycles. AC = air cooled at 8 °C/sec.

Figure 75 Microstructures after cyclic heat treatment at 1340 °C: a) 3 cycles; b) 6 cycles; c) and d) SEM BEI of a) and b) respectively. AC = air cooled at 8 °C/sec. Arrows indicate examples of γ grains.

Figure 76 Microstructure after cyclic heat treatment for 12 cycles at 1330 °C for 10 minutes followed by AC at 8° C/sec: a) optical image; b) BEI.

Figure 77 Effects of cyclic heat treatment temperature and cycles on lamellar grain size and volume fraction of equiaxed γ grains.

$\frac{1 \mu\text{m}}{(\text{c})}$

Figure 78 TEM photos of lamellae after cyclic heat treatment for 12 cycles at 1335 °C/10 min./AC: a) coarse γ lamellae γ_1 , γ_2 , γ_3 ; b) coarse γ lamellae and bundles of finer $L(\gamma/\alpha_2)$ lamellae; c) diffraction patterns for γ_1 , γ_2 , γ_3 showing they have the same crystallographic orientation, and for γ lamellae in $L(\gamma/\alpha_2)$ showing they have various orientations.

Figure 79 Schematics of cyclic heat treatment schedules with different heating and cooling rates.

Figure 80 Effects of cyclic heat treatment heating and cooling rates on microstructure, optical and BEI: a,b) 12 reference (RF) cycles; c,d) 12 slow heat (SH) cycles; cont'd on next page.

Figure 80 (cont'd) e,f) 12 slow cool (SC) cycles; g,h) 11 RF cycles + 1 SC cycle; i,j) 11 RF cycles + 1 rapid cool (RC) cycle; k) TEM of a γ/α_2 bundle in i,j.

Figure 81 Effect of cyclic heat treatment on microstructure: a) after heat treating at 1430 °C for 20 minutes, followed by water quench; b) specimen a) + 12 cycles/1335 °C/10 min./AC; c) same treatment as in a) but AC; d) specimen c) + 12 cycles/1335 °C/10 min./AC.

Figure 82 Microstructure of sample that was heat treated at 1370 °C/1 hr/WQ and then cyclically heat treated for 6 cycles/1335 °C/10 min/AC: a) optical image; b) higher magnification of windowed area in a); c) BEI of typical feathery lamellar colonies in a), dotted lines indicate apparent outlines of colonies.

Figure 83 a) Room temperature tensile curves for different microstructures: DPLX= duplex; FGFL= fine-grained fully lamellar; FL= fully lamellar; b) Hall-Petch plot of 0.2 % yield stress versus grain size parameter " $d^{-0.5}$ ".

Figure 84 Fracture surfaces of tensile specimens: a) fully lamellar, g.s. 300-600 μm ; b) fine-grained lamellar, g.s. 100-150 μm ; c) fine-grained fully lamellar, g.s. 50-70 μm ; d) duplex, g.s. 10-35 μm ; e) translamellar fracture in fully lamellar specimen showing brittle fracture mode and multiple crack formation on lamellae.

Figure 85 Deformation substructures in room temperature tensile specimens in: a, b) fully lamellar condition showing non-uniform distribution of dislocations and mechanical twins; c,d) fine-grained fully lamellar condition showing more uniform distribution of dislocations in the lamellae and mechanical twins across the lamellae.

Figure 86 Creep properties of different microstructures: a) time versus engineering strain; b) creep strain versus strain rate. DPEQX= dual-phase equiaxed; DPLX= duplex; FGFL= fine-grained fully lamellar; FL= coarse-grained fully lamellar.

Figure 87 Grain size vs minimum creep strain rate for different microstructures: DPEQX= dual-phase equiaxed; DPLX= duplex; FGFL= fine-grained fully lamellar; FL= fully lamellar.

Figure 88 Longitudinal sections of duplex creep tested specimen near the fracture surface, arrows indicate: a) secondary cracks developed below the fracture surface; b) examples of heavily distorted γ grains and lamellar grains.

Fracture Surface

↑
↓
Loading Direction

200 μm

Figure 89 Longitudinal section of fully lamellar creep tested specimen near the fracture surface. Arrows indicate intergranular cracks.

Figure 90 Longitudinal sections of fine-grained fully lamellar creep tested specimen showing: a) secondary cracks near the fracture surface; b) arrows indicate "soft" orientation grains; c) shear bands developed in a "hard" orientation grain.

Figure 91 TEM photos of creep deformation substructures in specimens with: a) and b) duplex microstructure, arrows indicate the formation of subgrain boundaries.; c) and d) fully lamellar microstructure, arrows indicate lamellae that are relatively free of dislocations.

(a)

(b)

Figure 92 TEM photos of creep deformation substructures in specimens with a fine-grained fully lamellar microstructure.

Figure 93 Schematic of continuous cooling transformation diagram: γ_s = gamma start; L_s = lamellar start; γ_{ms} = massive gamma start; after [61,62,64].

APPENDIX

Crucible Compaction Metals, Certificate of test for titanium powder Ti-47.5Al-3Cr, job number 9958A, lot 570, 28 May 1992.

CERTIFICATE OF TEST

Job Order No.: 9958 A

Customer NATIONAL RESEARCH COUNCIL CANADA	Purchase Order No. 31053-1-3586/01-0TT	Heat No. 570
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Product Description

Item No.	Pieces	Weight	Description
1	1	30.0 lb	Induction Melted/Gas Atomized Titanium Powder Ti-47.5Al-3Cr (at%), Lot 570 -35 US Std. Mesh.

HEAT 570 CHEMICAL ANALYSIS (wt%)

	Al	Cr	Fe	C	N	O	H	Ti	
wt%	33.4	4.0	.020	.014	.004	.092	.0030	Bal.	
%	47.1	2.9	.013	.044	.011	.219	.113	49.6	

SIEVE ANALYSIS (% Under)

									TAP DENSITY	FLOW RATE
US Std. No.	35	45	60	80	100	140	200	325	(g/cc)	(sec)
Microns	500	354	250	177	149	105	74	44	-35 mesh	-35 mesh
Heat 570	100	92	76	56	36	21	11	3	2.56	36

Sworn to and Subscribed Before Me

Certified By:

This 28th Day of May 1992M.C. Maranian Date: 5/28/92Title: QC Foreman

Notarial Seal
Judith K. Turek, Notary Public
North Fayette Twp., Allegheny County
My Commission Expires Nov. 6, 1993

Member, Pennsylvania Association of Notaries

IMAGE EVALUATION TEST TARGET (QA-3)

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