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**Microstructure Modification and Mechanical Behaviour of
an Investment Cast Near γ -TiAl Intermetallic Alloy**

by

Weijie Chen, M.S., B.S.

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

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

**Carleton University
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September 2000

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ABSTRACT

This thesis focuses on the development of a uniform lamellar structure with controlled minor phase precipitation in an investment cast Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si (at%) near γ -TiAl intermetallic alloy, and on the mechanical behaviour of this alloy with different microstructural conditions.

It is proven difficult to produce a uniform lamellar structure with relatively fine grains through a simple heat treatment, due to the coarse and non-uniform as-cast structure and non-uniformly distributed alloying elements along the cast bar. However, a rather uniform fully lamellar structure can be obtained by a homogenization and solution heat treatment, followed by a furnace cooling + air cooling two-step cooling technique.

Aging the lamellar structure at intermediate temperatures produces precipitate particles predominantly along α_2/γ lamellar interfaces or within α_2 lamellae, most of which are W- and Mo-rich β particles. Silicide precipitation is rather limited, compared to the β particles. The morphology of β particles obtained below 850°C is quite different from that at higher aging temperatures, and it is suggested that β particles form at below 850°C via a precipitation process controlled by interfacial diffusion of W and Mo. At higher temperature the formation of the β particles is initially controlled by short range diffusion of W and Mo. The activation energy for β precipitation below 850°C is about 366 kJ/mol.

The fully lamellar structure developed has decreased tensile strength and ductility at both ambient and elevated temperatures but an enhanced creep resistance, compared to the cast duplex structure. Aging the lamellar structure leads to an increased tensile strength, although the strength of aged lamellar structure is still lower than that of the cast duplex structure. Furthermore, the aged lamellar structure has an enhanced resistance to primary creep but a reduced creep life. The activation energy for creep of the lamellar structures is about 390 kJ/mol.

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LIST OF SYMBOLS

α	Ti ₃ Al phase with a hexagonal structure.
α_2	Ti ₃ Al phase with a DO ₁₉ hexagonal superlattice structure.
β	an ordered body centred cubic structure.
γ	TiAl phase with an L1 ₀ ordered face centered tetragonal structure.
γ_M	massive transformed γ structure.
γ_T	twinned γ structure.
γ_W	Widmanstätten γ structure.
σ	stress.
σ_f	friction stress.
$\sigma'_{i,i}$	friction stress component for element i corresponding to the interior of each grain.
$\sigma''_{i,i}$	friction stress component for element i compatible with neighbouring grains.
σ_{loc}	local stress.
$\dot{\epsilon}$	strain rate.
$\dot{\epsilon}_i$	strain rate of lamellar grain with specific orientation.
$\dot{\epsilon}_{min}$	minimum or steady-state creep strain rate.
A	projected area.
b	Burgers vector.
D	solute diffusivity.
F_i	volume fraction of lamellar grain with specific orientation.
K	microstructure-dependent constant.
n	stress exponents.
n'	stress exponents.
Q	activation energy.
R	universal gas constant.
S	activation area during creep.
t	time.
T	absolute temperature.
T_α	α transus temperature.
T_e	$\alpha \rightarrow \alpha_2 + \gamma$ eutectoid temperature.

1 Introduction

The objective of this thesis is to obtain a comprehensive understanding of the tensile and creep behaviour of an investment cast near γ -TiAl intermetallic alloy, Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si (at%), namely TiAl-WMoSi alloy, in the fully lamellar condition. It is anticipated that this understanding will support anticipated future applications of investment cast near γ -TiAl. The thesis research was conducted in collaboration with the Structures, Materials and Propulsion Laboratory, Institute for Aerospace Research, National Research Council Canada, from April 1996 to March 2000.

Near γ -TiAl intermetallics are being considered as potential materials for aerospace applications due to a unique combination of low density, good high temperature strength, stiffness retention, and thermal expansion that compares favourably to current alloys. As well, oxidation resistance and hot corrosion resistance are comparable to, or better than, those of current high temperature materials [1]. This combination of properties is attractive for several turbine and compressor component applications. Use of near γ -TiAl intermetallics for low pressure turbine blades would translate to weight savings [2], and for aerofoils in high temperature compressor stages, would lead to improved compressor performance [3].

Some engine components, such as turbine blades, made by casting are more competitive than those made by wrought or powder metallurgy methods due to the low cost of the casting process [4,5]. However, as-cast titanium aluminides have unacceptably low ductility and strength for many applications, due to the coarse and non-uniform structure formed during casting, that is not readily altered by hot isostatic

pressing (HIPing) [6]. Efforts to develop a more uniform microstructure have been made in some cast TiAl alloys [7] and it was suggested that TiAl alloys with refined microstructure may have the best balance of creep, tensile and fracture toughness properties [8]. Thus, the first objective of this thesis is to eliminate the coarse and non-uniform cast structure and to develop a uniform fully lamellar microstructure by using heat treatment. The research related to modification of the cast microstructure is presented in chapter 5.

Minor phase precipitation is believed to enhance creep resistance [9-12] and high temperature strength [13] by retarding dislocation motion at elevated temperatures. Precipitate particles have been characterized in several TiAl alloys containing W and Si [13-17], but, detailed analysis of the precipitation process has not previously been documented. Therefore, the second objective of this thesis is to develop an understanding of the precipitation kinetics and particle morphology in this near γ -TiAl alloy. This research is presented in chapter 6.

Some preliminary investigations of the properties of this alloy have been made, and the results show that the addition of alloying elements, Nb, W, Mo and Si, significantly improves tensile strength and creep resistance [18-20]. However, the mechanisms responsible for these improvements are not well understood, although it is generally believed that precipitates rich in W and Mo can retard the dislocation motion, thereby hardening the material [20]. Furthermore, the tensile and creep properties of this alloy have only been evaluated for the as-cast and homogenized conditions, whereas the influence of fully lamellar microstructure and precipitation particles on the tensile properties and creep resistance of this alloy has not been investigated systematically.

Therefore, the last objective of this thesis is to determine the tensile and creep behaviour of this alloy as a function of microstructures and to explain the mechanism of creep through examination of deformed structures. This research is presented in chapter 7.

Hence, this thesis work will

- i) modify the non-uniform cast microstructure to obtain a uniform fully lamellar structure.
- ii) develop controlled precipitate particles within this fully lamellar microstructure and analyze the precipitation process,
- iii) investigate the tensile and creep behaviour of the TiAl-WMoSi alloy as a function of microstructure, and
- iv) evaluate the creep deformation processes.

From the thesis work, an enhanced understanding will be generated in the following areas:

- i microstructural response to heat treatment of investment cast γ -TiAl alloys,
- ii precipitation process in γ -TiAl alloys,
- iii effect of microstructure on tensile and creep behaviour,
- iv effect of precipitation on tensile and creep behaviour, and
- v creep processes in the developed microstructures.

This thesis will contribute to the knowledge required to assess investment cast near γ -TiAl intermetallic alloys for high temperature turbine blade applications. The technology developed in this thesis will also be useful for the future development of near γ -TiAl intermetallic alloys.

2 Literature Review

Low density, high melting temperature, good elevated-temperature strength and modulus retention, high resistance to oxidation and hydrogen absorption and excellent creep properties of gamma base titanium aluminide alloys make them potential candidate structural materials for high temperature gas turbine aero-engine applications [21-26]. Since the first major gamma alloy development program initiated in the 1970's, extensive work has been conducted on alloy development, microstructural modification and property evaluation [6,22,24-25]. Most of the properties of gamma base titanium aluminides, when adjusted for density, appear to be comparable to, or better than, those of the counterpart nickel-based superalloys for which they may be substituted [6]. In addition, cast gamma based alloys are more competitive than those made by wrought or powder metallurgy methods due to the lower cost of the casting process [4,5]. However, as-cast titanium aluminides have unacceptably low ductility and strength for many applications, due to the coarse and nonuniform cast lamellar structure that is not readily altered by hot isostatic pressing (HIPing) [6]. Efforts to control the lamellar structure through heat treatments have been made in the past few years. In light of the foregoing and the emphasis of this thesis, the following literature review concentrates on the microstructure – mechanical property – deformation interrelationship for γ -TiAl alloys. To fully appreciate this interrelationship a brief overview of the physical metallurgy of γ -TiAl alloys is first presented.

2.1 Phases and structures in gamma base TiAl alloys

Two major phases, γ -TiAl phase and α_2 -Ti₃Al phase, exist in gamma base binary TiAl alloys, as shown in Fig. 2.1 [27]. Some other minor phases may also exist in this type of alloy if additions of alloying elements are made.

2.1.1 Phase components and their crystal structures

The γ -TiAl phase has an L1₀ ordered face centered tetragonal structure (Fig. 2.2a), with a wide range of compositional stability from 48.5 at% Al to 66 at% Al [23]. The α_2 -Ti₃Al phase has a DO₁₉ hexagonal superlattice structure (Fig. 2.2b), with a wide composition range of 22-39 at% Al. With the additions of alloying elements Cr, Nb, W, Mo and Si, minor phases such as β , ω and titanium silicides may also appear in gamma base TiAl intermetallics.

The β phase, having an ordered body centred cubic structure (namely B2 structure), will exist with the addition of β stabilizing elements such as Cr [28-31], Nb [32-34], W [14,35,36], Mo [37-41] and Zr [39]. The β (B2) phase is always rich in these elements. The ω phase, with an ordered hexagonal structure [14,30,32,33,38], usually exhibits a strong orientation relationship with β phase in γ base intermetallics containing these elements (Fig. 2.3). Titanium silicides, usually in the form of Ti₅(AlSi)₃ [9,15-17], have a complex hexagonal structure [15,16]. Laves phase, rich in Mn, Cr and Ta and having a hexagonal structure (TiMn₂ type), is also observed in some near γ -TiAl alloys [42,43].

2.1.2 Microstructures of near γ TiAl alloys

When near γ TiAl alloys, of the composition 43 – 49 at% Al, are cast or air or furnace cooled from equilibrium at a temperature in the α phase region (Fig. 2.1), the resulting microstructure has a lamellar morphology (Fig. 2.4a) or a nearly lamellar morphology, with a very small amount of fine equiaxed γ grains existing at the lamellar grain boundaries (Fig. 2.4b). γ -TiAl plates and α_2 -Ti₃Al plates are well aligned in the lamellar regions of these two structures [24,44]. When heat treated in the $\alpha + \gamma$ phase region, a duplex microstructure consisting of lamellar grains and γ grains forms (Fig. 2.4c). A structure without lamellar constituent in it is referred to as equiaxed γ structure (Fig. 2.4d). When a lamellar structure is heat treated in the $\alpha_2 + \gamma$ region, a duplex microstructure consisting of both lamellar grains and γ grains can also be obtained. The formation of γ grains is sluggish in alloys with aluminum concentration of 48 at% or lower, so it can be avoided during casting or cooling from the α phase region.

The cooling rate, after heat treatment in the α phase region, has a very strong influence on the microstructure of near γ -TiAl alloys. A lamellar structure can form only at a low cooling rate [45-47], with the lamellar interface spacing decreasing as cooling rate increases [44]. At intermediate cooling rates, both Widmanstätten γ (γ_w) and feathery morphologies appear, and at high cooling rates a massive transformed structure (γ_M) occurs (Fig. 2.5). Generally speaking, as the cooling rate increases after heat treatment in α region, the microstructure changes according to the following sequence: lamellar $\rightarrow \gamma_w + \gamma_M + \text{lamellar} \rightarrow \gamma_w + \gamma_M \rightarrow \gamma_M \rightarrow \gamma_M + \alpha_2$ (Fig. 2.6) [46]. As the cooling rate further increases, the volume fraction of α_2 increases in the $\gamma_M + \alpha_2$ structure, and an

equiaxed α_2 structure is obtained after cooling from α phase region at a cooling rate of about 2000°C/s [48].

After heat treatment in the α phase region, the cooling rate also strongly affects the lamellar grain boundary morphology [49-51]: higher cooling rates cause planar grain boundaries while lower cooling rates lead to serrated grain boundaries with interlocked lamellae along these boundaries. The extent of the discontinuous coarsening of lamellar structures at grain boundaries, as a result of a lower cooling rate, produces serrated/interlocked lamellar grain boundaries, due to the higher $\alpha \rightarrow$ lamellar transformation temperatures and the larger driving forces for γ formation [49].

For the minor phases, if β stabilizers are added, the β (B2) phase (Fig. 2.7a) forms during casting with a blocky shape, which exists along the cast lamellar grain boundaries or within the lamellar grains [14,19]. In silicon-containing cast alloys, titanium silicide particles (Fig. 2.8a) can be observed in both equiaxed γ and lamellar grains [16,20]. Heavy alloying elements, such as W, usually segregate toward the dendrite core regions [36], while cast B2 and silicide particles segregate to the interdendritic regions, grain boundaries [14] and last solidified regions [52]. These cast B2 and silicides may dissolve into the α phase during thermal exposure in the α phase region however, segregation of tungsten still remains [14,36], due to the low diffusivity of W [36,53]. Excessive thermal exposure at high temperature is required to eliminate W segregation [36].

2.1.3 Precipitation in near γ -TiAl alloys

After annealing at relatively low temperatures, below the eutectoid temperature (Fig. 2.1), the β phase forms as blocks [14,28,33,38], rods [14] and fine needles [14,35],

as shown in Fig. 2.7b and 2.7c. It was suggested that blocky-shaped β derived from segregation during solidification, whereas rod-like β formed from lamellar coarsening and needle-shaped β phase precipitated during annealing [14]. Needle-shaped β particles usually precipitate inside α_2 plates and at the α_2/γ interfaces [14,35]. β particles along lamellar/ γ boundaries in duplex structure, formed during annealing, have semi-coherent interfaces with both the parent α_2 and the adjacent γ grains [54]. Fine titanium silicides (Fig. 2.8b), formed during annealing, appear in α_2 , γ and β phases preferably at α_2/γ , γ/γ and β/γ interfaces, but mostly at α_2/γ and β/γ boundaries [9,15-17] and grow coherently into α_2 and β matrix phases [15]. In some β (B2)-containing TiAl alloys, fine lens-shaped ω particles precipitate inside the β blocks [14,30], after thermal exposure at elevated temperatures. The Laves phase usually precipitates along the lamellar grain boundaries and α_2/γ and γ/γ interfaces [43] but only in a typical TiAl alloy. Aside from β and silicide precipitation, some other fine precipitate particles such as borides [55], carbides [10,13,17] and nitrides [10] exist in near γ -TiAl alloys with the addition(s) of boron, carbon and/or nitrogen.

2.2 Microstructure and property relationships in near γ -TiAl alloys

Good high temperature strength retention, high fracture toughness, good oxidation resistance and good creep resistance are some of the major requirements for structural materials for high temperature gas turbine aero-engine applications, while adequate room temperature ductility is necessary for manufacturing and assembling. A good balance among strength, fracture toughness, creep resistance and room temperature ductility should be established for near γ -TiAl.

2.2.1 Effect of microstructure

As mentioned in section 2.1.2, four different microstructures (Fig. 2.4) can be obtained with near γ -TiAl intermetallics. Each of these microstructures yields a distinct set of mechanical properties. In general, the equiaxed γ structure has the highest tensile strength [56]. Tensile strength decreases as the volume fraction of lamellar constituent increases, with the lowest tensile strength exhibited by the fully lamellar structure (Fig. 2.9). Nonetheless, decreasing the grain size of the fully lamellar structure results in an increase in the strength, and a fine-grained fully lamellar structure may have a strength level higher than that of a duplex structure [57]. Both equiaxed γ and fully lamellar structures have poor ductility [25,56], and the best ductility is achieved by the duplex structure (Fig. 2.9). The duplex structure has lower fracture toughness than the lamellar structure (Fig. 2.10) [25]. In addition, near γ -TiAl alloys with microstructures of large lamellar grains yield lower ductility and fracture toughness [25], compared to lamellar structures of small grain size.

The tensile behaviour of lamellar grains is very anisotropic. Lamellar grains with α/γ interfaces perpendicular or parallel to the stress direction ("hard-oriented" grains) have higher strength and lower ductility [58-60], while those with lamellar interfaces inclined from the stress direction ("soft-oriented" grains) have lower strength but higher ductility (Fig. 2.11). The poor ductility of hard-oriented lamellar grains is attributed to the shear deformation across the lamellae [61], since the slip traces in these lamellar grains proceed mainly on $\{111\}$ planes across lamellae [62]. On the other hand, deformation of soft-oriented lamellar grains occurs mostly on $\{111\}$ planes parallel to the lamellar interfaces [62] and therefore, the increased ductility of soft-oriented lamellar

grains can be associated with the shear deformation parallel to lamellar planes [61]. Large strain discontinuities at boundaries between large lamellar grains, due to the anisotropic stresses between the grains, may lead to crack initiation at the boundary, resulting in low ductility of near γ -TiAl with only large lamellar grains. As such, near γ -TiAl alloys with fine-grained fully lamellar structure having small lamellar interface spacing may have the best balance between tensile properties and fracture toughness [8].

The creep resistance of near γ -TiAl alloys increases with the increased volume fraction of lamellar grains, and a near γ -TiAl becomes creep resistant only when the lamellar fraction exceeds 80% (Fig. 2.12) [56,63,64]. It is widely acknowledged that the fully lamellar structure is the most resistant to creep [56,63-71]. Furthermore, fully lamellar structures with finer lamellar interface spacing have better creep resistance (Fig. 2.13) [50,51,72-75], since lamellar interfaces serve as obstacles to dislocation motion. However, microstructure degradation may take place at intermediate temperatures, typically 800°C – 1200°C, and discontinuous coarsening of lamellae is more substantial in the finer lamellar material [76], leading to premature failure. One report revealed that extended thermal exposure at intermediate temperature reduces creep life but the reason was undetermined [77]. Fine lamellar structures with Widmanstätten and feathery features, formed at relatively high cooling rate, have lower creep resistance [50,73], which was attributed to the planar grain boundaries and/or the anti-phase domain boundaries (APBs) formed within lamellae. A serrated grain boundary morphology with interlocked lamellae along grain boundaries will effectively prevent lamellar grain boundaries from sliding during the creep [78].

In summary, the duplex structure is desirable for ductility and strength, adequate for creep resistance, and poor for fracture toughness, while the fully lamellar structure is more desirable for toughness, attractive for creep and poor for ductility and strength [79]. A balanced set of these properties can be expected by reducing lamellar grain size and lamellar interface spacing.

The presence of blocky β (B2) phase along the lamellar grain boundaries or inside the lamellar grains will strengthen near γ -TiAl [80], however, it reduces room temperature ductility [81] since the β phase acts as a source of micro-cracking at grain boundaries [41]. However, in contrast, β phase along grain boundaries elongates during deformation resulting in large ductility at elevated temperatures [82,83]. Blocky β (B2) phase is considered detrimental to both room temperature tensile properties [41] and creep resistance [19,84].

Fine precipitate particles, such as β (B2), titanium silicide (Ti_5Si_3), carbide (Ti_2AlC) and nitride (Ti_2AlN), may retard dislocation motion at elevated temperatures thereby enhancing creep resistance [10-12,42] and high temperature strength [13].

Thus, a balanced set of high temperature strength, fracture toughness, creep resistance and room temperature ductility may be achieved by producing a stable fully lamellar structure with (1) small grain size, (2) serrated/interlocked grain boundary morphology, (3) fine lamellar interface spacing and (4) fine precipitates along lamellar interfaces [44].

2.2.2 Effect of alloying elements

Attention has been addressed to the effect of alloying elements on mechanical properties and, in general, it is acknowledged that elements such as V, Cr, and Mn will increase the ductility of $\alpha_2 + \gamma$ TiAl intermetallics. Nb, W and Ta may enhance the resistance to deformation and thereby decrease the ductility [22], whereas the addition of Mo does not significantly affect the ductility [85,86]. However, excessive addition of Cr seems to be deleterious to ductility, due to the formation of the blocky β (B2) phase [81]. The intrinsic strengthening effect of Cr, W, Mo and Nb [81,83,86-88] is not well understood, due to the overlap of the microstructural factors such as grain size, orientation of lamellae, etc. [62]. Meanwhile, the effect of alloying elements may overlap with those due to the manufacturing method, for example, tungsten strengthens near γ -TiAl alloys produced by the powder metallurgy technique. However, it significantly reduces the strength of TiAl produced by ingot metallurgy [36], which may be the result of microstructural inhomogeneity in the ingot metallurgy material.

Addition(s) of C, B, N and Si can also increase the strength of near γ -TiAl intermetallics [89]. Small amounts of silicon will strengthen this type of alloy [86,90,91]. However, an excessive amount of silicon will diminish the strengthening effect [90,91], due to the coarse Ti_5Si_3 particles that initiate early failure via particle debonding and particle cracking.

The intrinsic effect of alloying elements on the creep behaviour of near γ -TiAl intermetallics is also not yet well understood, since investigations of these effects are usually influenced by many other factors such as processing, microstructure, test condition and so on. It is known, in general, that alloying elements having a positive

effect on high temperature strength and resistance to deformation, such as Nb [29], Mo [86], Ta [29] and Zr [92] will enhance the alloy's creep resistance. Alloying elements that increase the ductility, such as Cr and Mn [86,93] will be detrimental to creep resistance. Once again, W [35,36,53,94] enhances the creep resistance of powder metallurgy near γ -TiAl alloys but it decreases the creep resistance of an ingot metallurgy alloy [36].

Addition of silicon will result in the formation of silicide particles and consequently an increase in creep resistance [9,95,96] by pinning dislocation segments. However, such an effect may be eliminated if the alloy contains an excessive amount of silicon (Fig. 2.14), due to the dynamic recrystallization during creep deformation caused by a large amount of silicon [95]. It was suggested that excessive addition of silicon result in an increase in volume fraction of the Ti_5Si_3 phase due to the consumption of the α_2 phase, leading to a decrease of the α_2/γ lamellar structure [95]. On the other hand, small additions of silicon may increase the mobility of interfacial dislocations [87] thereby decreasing the creep resistance of near γ -TiAl alloys [97], if silicon acts as a solution element in the matrix. Interstitial elements, such as C and N [10,98] also increase the resistance to creep in near γ -TiAl alloys, probably by forming precipitate particles.

The increased creep resistance due to the addition of alloying elements is mainly due to two processes: precipitation strengthening and/or solid solution strengthening. It is found that Nb, Mo, W, Ta, and Zr enhance the creep resistance of near γ -TiAl alloys regardless of whether they are part of precipitate particles or remain as solid solution elements, but Si will enhance the creep resistance only as part of precipitate particles.

Addition of tungsten improves the creep resistance of near γ -TiAl intermetallics to the largest extent, due to the low diffusivity of W solute and β precipitation at grain boundaries and lamellar interfaces [11,53]. Moreover, W will segregate to both γ/γ and α_2/γ interfaces within the lamellar structure, thereby stabilizing the α_2 lamellae against dissolution and coarsening during thermal exposure [99].

Addition of boron refines the lamellar structure [100-102] and investment cast near γ -TiAl alloys have fine grained uniform cast microstructures when TiB_2 particles are embedded in the material [103]. However, boron alone may cause fragmented and discontinuous α_2 lamellae [100] and the formation of equiaxed γ grains [102] during subsequent aging, making the fully lamellar alloys containing boron unstable at intermediate temperatures, compared to the alloy with additions of both W and B.

2.2.3 Lamellar grain refinement and the stability of lamellar structure

As mentioned in section 2.2.1, near γ -TiAl intermetallics with a fine-grained fully lamellar structure may have a good balance of properties for high temperature applications. The grain size of lamellar structures can be reduced to 50 – 150 μm by thermo-mechanical processing (TMP) such as isothermal forging, hot die forging and hot-extrusion [104,105] at temperatures near the α transus temperature (T_α). Thermo-mechanical treatment (TMT), a process comprised of TMP followed by a heat treatment near T_α [105], can produce a lamellar structure with small grain size of 100 – 300 μm . In addition, a fine-grained fully lamellar structure of about 50 μm in size can also be obtained by annealing the cast lamellar structure at temperatures below T_α for a long time followed by a brief heat treatment in the α phase field [7]. Annealing in the $\alpha + \gamma$ phase

region will result in the formation of a near-gamma structure owing to the discontinuous coarsening of α_2 lamellae [7,106]. Moreover, a cyclic heat treatment at about the α transus temperature (T_α) was developed to obtain a fine-grained lamellar microstructure [107].

The fine lamellar structure has favourable mechanical properties. However, this type of microstructure may be unstable [99,104,108] during thermal exposure at intermediate temperatures (between 700°C and 1000°C), due to discontinuous coarsening and decomposition of the α_2 lamellae. Meanwhile, thermal exposure under stresses at intermediate temperatures will cause discontinuous coarsening of α_2 lamellae [76], dynamic recrystallization of the single-phase gamma grains at lamellar colony boundaries and spheroidization of the α_2 -Ti₃Al laths [109-114]. The instability of lamellar structures can lead to the early failure of the material. The addition of a small amount of W results in interfacial segregation of W, thereby stabilizing the α_2 lamellae against growth and coarsening during aging at intermediate temperatures [99].

2.3 Deformation of near γ -TiAl intermetallics

The movement of dislocations, along with the formation of deformation twins, assists the deformation of near γ -TiAl intermetallics [38,87,90,115-118]. Most deformation defect structures appear within γ grains and γ lamellae. Much less deformation takes place in the α_2 phase, due to its limited slip systems. The features of plastic deformation at ambient and elevated temperatures are quite different, leading to significant differences in ductility of the near γ -TiAl intermetallics.

2.3.1 Deformation at room temperature

Plastic deformation by slip and twinning within a grain for both duplex and lamellar structures is planar [115,119] at room temperature. Most of the dislocations contributing to plastic deformation are of the $1/2\langle 110 \rangle^*$ type gliding on $\{111\}_\gamma$ planes, with very few $\langle 101 \rangle$ super dislocations on the same planes in the γ areas [38,115,117]. Some superlattice dislocations with Burgers vector of $1/2\langle 112 \rangle$ were observed in both polysynthetically twinned crystals (PST TiAl) and polycrystalline TiAl [115,118]. Partial dislocations with Burgers vector of $1/6\langle 112 \rangle$ were also observed in polycrystalline TiAl [115,120]. When silicon is in solution, the mobility of $1/6\langle 112 \rangle$ partial dislocations is increased which consequently enhances room temperature ductility [87]. The formation of deformation twins is another mechanism for deformation of near γ -TiAl. The deformation twins are of $\{111\}\langle 11\bar{2} \rangle$ type [38,116-118,120], and may form via generation and slip of $1/6\langle 112 \rangle$ partial dislocations in the $\{111\}_\gamma$ plane [116,120]. Movement of both $\langle 100 \rangle$ and $\langle 111 \rangle$ type dislocations can be expected in the β (B2) phase, while very few $\langle 11\bar{2}0 \rangle$ dislocations can be found in α_2 lamellae [38,121]. The α_2 phase is the least deformable due to the limitation of slip systems imposed by the compatibility of deformation with the γ and β (B2) phases [38].

The deformation within lamellar grains is anisotropic (Fig. 2.11), as a result of the orientations of lamellar interfaces with respect to the stress directions. In the γ lamellae of "soft-oriented" grains, deformation proceeds predominantly via the slip of $1/2\langle 110 \rangle$ dislocations and some $1/2\langle 112 \rangle$ superlattice dislocations, with some deformation twinning [118]. In the γ lamellae of "hard-oriented" lamellar grains, both mechanical

* $\langle hkl \rangle$ denotes a family of directions in the ordered γ (tetragonal) structure. The h and k indices can assume all allowed values but the l indices cannot change.

twins and $1/2\langle 110 \rangle$ dislocations assist the deformation with $\{111\}\langle 11\bar{2} \rangle$ deformation twins being the principal deformation mode [118,122]. No significant change in deformation structure of α_2 lamellae was observed, due to the limited slip systems in this crystal structure.

In the lamellar structure, deformation starts in the “soft-oriented” lamellar grains well below the yield stress levels and then propagates into the adjacent grains, usually in a “hard-oriented” lamellar grain [57], through the buildup of stress concentrations at grain boundaries. The slip transfer from γ to α_2 in the “hard-oriented” lamellar grains is difficult and usually leads to cleavage fracture at the γ/α_2 interfaces [57]. However, transfer of deformation across the lamellar interfaces is possible when γ deforms by twinning [121] (Fig. 2.15) and/or if the fully lamellar grain is small (Fig. 2.16). In lamellar structures with a large grain size, increased stress concentrations at lamellar grain boundaries due to dislocation pile-ups via the deformation along lamellar interfaces in “soft-oriented” lamellar grains will lead to nucleation of microcracks (Fig. 2.17) in the adjacent “hard-oriented” lamellar grain [119] and therefore lower the ductility.

2.3.2 Deformation at elevated temperatures

At elevated temperatures, the mobility of $\langle 011 \rangle$ superdislocations and $1/6\langle 11\bar{2} \rangle$ partial dislocations in the γ phase increases rapidly [117], contributing to high temperature deformation, in addition to the contributions from previously mentioned sources. Both dislocations and mechanical twins assist the deformation during creep [66,72,109,123,124]. Again, deformation at elevated temperatures shows anisotropic features [125], with deformation twins mostly appearing within the “hard-oriented”

lamellar grains (Fig. 2.18). Fewer mechanical twins are observed when material being crept at relatively higher temperature [126] and, the number of deformation twins increases when the applied stress is increased. During creep deformation, both lamellar interfaces and precipitation particles act as obstacles to dislocation motion (Fig. 2.19) [127]. As the deformation proceeds, deformation transfer across the lamellae takes place (Fig. 2.20a) accompanied by void formation [65,69,125]. At the same time, the lamellar structure becomes unstable and α_2 coarsening and dynamic recrystallization occur (Fig. 2.20b & c) [109-114].

2.3.3 Mechanisms of deformation

The details of the deformation mechanisms remain unclear, since most of the observations of the deformation structures are undertaken after deformation. However, an *in situ* study shows that at room temperature, only the interfacial dislocations in twin-related (γ/γ_1) interfaces are mobile [128], suggesting that the critical resolved shear stress for the γ/α_2 interfaces is much higher. The limited mobility of dislocations at room temperature leads to poor ductility of near γ -TiAl intermetallics.

Many observations show dislocations bowed out from α_2/γ interfaces of the lamellar structure when deformation takes place at elevated temperatures [125,127,129], suggesting that interfacial dislocations are generated and emitted from α_2/γ interfaces and then glide within the γ lamellae. Screw dislocations in the interfaces are supposed to be the sources for emitting matrix dislocations [87]. An *in situ* observation reveals that upon heating, dislocation loops can be generated at the semicoherent α_2/γ interfaces (Fig. 2.21) in the lamellar areas and expand on $\{111\}_\gamma$ planes obliquely oriented to the interfaces

even in the undeformed condition [130]. The generation and subsequent motion of these dislocations lead to deformation of the lamellar structure. At elevated temperatures, cross slip on $\{111\}_\gamma$ plane (Fig. 2.22) becomes possible providing another possible dislocation source [87].

In summary, it is expected that at room temperature, dislocations will glide along γ/γ_1 interfaces in the "soft-oriented" lamellar grains at the beginning of deformation and, as the deformation increases, glide of dislocations at α_2/γ interfaces becomes possible. Once the dislocations move to the lamellar grain boundaries, the stress concentration at the boundaries increases as a result of dislocation pile-ups, which enable deformation to propagate into the adjacent "hard-oriented" grains through the formation of mechanical twins. However, owing to the difficulty for deformation to transfer across the α_2 plates, increased stress concentrations at lamellar grain boundaries, due to dislocation pile-ups via the deformation along lamellar interfaces in "soft-oriented" lamellar grains, will lead to nucleation of microcracks in the adjacent "hard-oriented" lamellar grain [119] (Fig. 2.17). The limited number of mobile dislocations as well as nucleation of microcracks result in fracture of near γ -TiAl at low strain levels at room temperature.

At elevated temperatures, dislocation glide becomes easier at both γ/γ_1 and α_2/γ interfaces and these interfaces act as dislocation sources. Additionally, the mobility of $\langle 011 \rangle$ superdislocations and $1/6\langle 11\bar{2} \rangle$ partial dislocations in γ areas increases significantly [115]. The increased mobility of $1/6\langle 11\bar{2} \rangle$ partial dislocations favours the formation of $\{111\}\langle 11\bar{2} \rangle$ mechanical twins [87] and consequently enhances deformation transfer across the α_2 lamellae [121]. At the same time, cross slip of dislocations across lamellar interfaces become possible, providing more dislocation sources during

deformation [87]. The increased number of mobile dislocations and deformation transfer across α_2 lamellae provides higher ductility at elevated temperatures. Decreasing lamellar interface spacing will increase the number of interfaces, consequently inhibiting dislocation motion and thus enhancing creep resistance.

2.4 Mechanisms of creep in near γ -TiAl intermetallics

Despite the large number of investigations, the mechanism of creep for near γ -TiAl is still not well understood. Previous research shows that two phase near γ -TiAl intermetallics exhibit a power-law behaviour, in which the minimum creep strain rate ($\dot{\epsilon}_{min}$) can be described by the Arrhenius equation:

$$\dot{\epsilon}_{min} = K\sigma^n \exp(-Q/RT) \quad \dots\dots (2-1)$$

where K is a microstructure-dependent constant, n the stress exponent, Q the activation energy for the creep process, T the absolute temperature, σ the applied stress, and R the universal gas constant. This equation is also referred to as the power law creep equation.

Creep with a stress exponent of about 1 is usually related to a diffusional creep mechanism, and dislocation climb controlled creep mechanism is, in general, associated with a stress exponent between 4 and 7. For two phase γ base alloys, the stress exponent varies from 0.6 to 3.5 [35,72,110,131,132] when crept at low stress levels. At high stresses, the stress exponent falls between 7 and 9, or even higher [9,35,65,72,123,131-136], leading to the supposition that creep at high stress levels is controlled by dislocation climb. At temperatures higher than 800°C, dynamic recrystallization of the equiaxed γ grains at lamellar grain boundaries and spheroidization of the α_2 laths may occur, which

will result in the loss of creep strength [109,114]. Under such circumstances, determination of the stress exponent is unlikely to provide information regarding the creep mechanism [44].

For pure metals, dislocation climb controlled creep is associated with a stress exponent between 4 and 7 [137-139], and the activation energy for creep is equal to that for self-diffusion [138]. In near γ -TiAl intermetallics the increase of stress exponent with increasing stress has been concluded to be the result of a transition from diffusional creep at low stresses [35,110,140] to dislocation climb controlled creep [35,109,110,134] or power law breakdown (dislocation glide controlled creep) [35,72] at high stresses. Stress exponents greater than 4.5 at high stress levels have been postulated [44] to be the result of various factors such as the friction stress within lamellar structures due to the small lamellar spacing [141] and structure instability [109], which need to be overcome before dislocations become mobile [136].

Analyses regarding the friction stress were conducted for the fully lamellar structure by modifying the equation (2-1) to

$$\dot{\epsilon}_{min} = K(\sigma - \sigma_0)^n \exp(-Q/RT) \quad \dots\dots (2-2)$$

where σ_0 is the friction stress. However, calculations revealed that friction stress occurs only at high stress levels [136,142], whereas in the low stress regime, friction stress is null or small. It was suggested that the lower stress exponent obtained in the low stress regime does not represent a change in dislocation mechanism, but only a change in the absolute value of the friction stress that needs to be overcome locally to initiate dislocation mobility [136]. Nevertheless, null friction stress was determined for the fully

lamellar structure crept at 760°C and below 150 MPa [142], suggesting that a change in creep mechanism might occur.

It has been noted that the substructure developed during creep is inhomogeneous and varies between grains as a function of the “hard” or “soft” orientation [72,125,136]. For such an inhomogeneous deformation, a proposed qualitative model [135] treats each grain individually, with the total strain rate given by

$$\dot{\varepsilon} = \sum F_i \dot{\varepsilon}_i \quad \text{..... (2-3)}$$

where F_i and $\dot{\varepsilon}_i$ are the volume fraction and the strain rate of each grain respectively. As such, accounting for the friction stresses within and between lamellar grains, the total strain rate is given by

$$\dot{\varepsilon} = \sum F_i K [\sigma - (\sigma'_{in} + \sigma''_{in})]^4 \exp(-Q/RT) \quad \text{..... (2-4)}$$

where σ'_{in} and σ''_{in} are the friction stress components for element i corresponding to the interior of each grain (σ'_{in}) and compatibility with neighbouring grains (σ''_{in}). A stress exponent of 4 corresponding to dislocation climb controlled creep can be expected for the power law relationship (equation (2-1)) only if $\sigma'_{in} + \sigma''_{in} = 0$. The effect of some microstructural parameters can be qualitatively explained using this model [135], such as the high σ'_{in} that results from small lamellar interface spacing and high σ''_{in} due to smaller grain size. However, this modified model may still have problems similar to those of equation (2-2), i.e. friction stress components will change as the stress changes.

The determined activation energy for creep of near γ -TiAl is between 180 – 450 kJ/mol [9,67,93,110,131-134,140,143-145], which may be dependent on stress [140] and increases with the volume fraction of lamellar constituent [145]. Most of these values are

much higher than those for interdiffusion, the diffusion between diffusion couples with different composition, and Ti self-diffusion in γ -TiAl phase. In γ phase, activation energy is 130 – 312 kJ/mol for interdiffusion [146-148], depending on the composition of diffusion couples, and 250 – 290 kJ/mol for Ti self-diffusion [149,150]. These suggest that the activation energies measured represent apparent activation energies, especially at high stress levels. It was therefore suggested that thermal energy could not be the only controlling mechanism for the creep [143]. Thus, considering the factor of thermally activated glide of dislocations during the creep, the apparent activation energy Q can be described [151]:

$$Q = Q_0 - Sb\sigma_{loc} \quad \dots\dots (2-5)$$

where Q_0 is the thermal energy (for diffusion), σ_{loc} is the local stress to help the thermal energy such that the thermally activated event can be made to occur, S is activation area and b is Burgers vector. The local stress, σ_{loc} , can only be measured from the dislocation substructures [152].

Based on the observed deformation structure after creep and the measured stress exponent, it is suggested that the creep of multiphase near γ -TiAl alloys be controlled by dislocation climb [63,134,153-155] within medium stress regime, and by intrinsic diffusion [140] at low stress levels. At high stresses, the creep of multiphase near γ -TiAl intermetallic alloys is controlled by the glide of dislocations [35,72].

2.5 Current understanding of near γ -TiAl with additions of W, Mo and Si

Near γ -TiAl with additions of W, Mo, and Si was found to have superior creep resistance to most other near γ -TiAl alloys [19,20,156], when the cast material was

subjected to a heat treatment at 1010°C for 50 hours. It was found that precipitates containing W, Mo and/or Si preferably occur in the deformed gage section during creep [156,157], and these precipitates are thought to nucleate during heat treatment and grow with the assistance of strain or consumption of α_2 [157]. The presence of these precipitates interferes with dislocation motion and also hardens the microstructure thereby enhancing creep resistance of the WMoSi alloy [156,157].

It was also noticed that the cast + HIPped material has a higher lamellar volume fraction and it crept much faster to 0.5% strain, compared to the 1010°C/50hr heat treated material. As such, it was suggested that a lower volume fraction of lamellar grains and the presence of precipitates account for the excellent primary creep resistance in the heat-treated alloy [157]. This result seems to be different from those in section 2.2.1 – that a fully lamellar structure may have the best creep resistance in near γ -TiAl. This apparent abnormality might be due to the higher strength of the cast structure with increased equiaxed γ grains (Fig. 2.9).

2.6 Summary

The near γ -TiAl intermetallics, with α_2 and γ phases as the major constituents, have four typical microstructures, namely: fully lamellar, near lamellar, duplex and equiaxed γ . The duplex structure is desirable for ductility and strength, adequate for creep resistance, and poor for fracture toughness, while the fully lamellar structure is more desirable for toughness, attractive for creep resistance and poor for ductility and strength. A balanced set of high temperature strength, fracture toughness, creep resistance and room

temperature ductility may be achieved by producing a fully lamellar structure with small grain size, fine lamellar interface spacing and serrated grain boundary morphology.

Additions of alloying elements may alter the mechanical properties of near γ -TiAl by forming minor phase precipitation particles such as β , silicides, carbides, borides, nitrides and Laves phase, or via solid solution strengthening. Precipitation particles will, in general, strengthen the material and enhance the creep resistance. Solute atoms such as W, Nb, Ta and Zr enhance the creep resistance, but Si seems to do the opposite. Microstructure instability is an issue to be addressed for application at elevated temperatures. It can be improved by the addition of alloying elements with low diffusivity.

Therefore, a stable fully lamellar structure with small grain size, serrated/interlocked grain boundary morphology, fine lamellar interface spacing and fine precipitation particles may have a favourable balance of high temperature strength, fracture toughness, creep resistance and room temperature ductility.

3 Experimental Procedures

This thesis research involves experimental work in metallography, electron microscopy, x-ray diffraction, heat treatment, tensile and creep tests. This section describes the details of the experimental procedures.

3.1 As-received alloy condition

The γ -TiAl alloy used for this research, namely TiAl-WMoSi alloy, has a nominal composition of Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si (at%). This alloy was developed by Howmet Corporation to improve creep resistance through W, Mo and Si additions. The chemical composition of the alloy is as follows:

Table 3.1 Chemical composition of γ -TiAl alloy

	Al	Nb	Mn	W	Mo	Si	Ti
atomic percentage (at%)	47.02	1.98	0.94	0.5	0.5	0.21	balance

The alloy was investment cast followed by hot isostatic pressing (HIPing) at 1260°C and 172.5 MPa for 4 hours, by the Howmet Corporation. The cast bars were 200 mm in length and 15 mm in diameter. The material supplied in as-cast plus HIPped condition.

3.2 Heat Treatment

Heat treatments were conducted to modify the non-uniform cast microstructure to a uniform fully lamellar structure. Samples of about 12 mm in length were cut from both the top (last solidified) and bottom (first solidified) sections of the cast bar, according to the geometry of the cast bars, to ensure the desired microstructure was obtained

throughout the entire bar. To avoid specimen oxidation, all the heat treatments were conducted in protective atmosphere by encapsulating samples in quartz tubing. The samples were wrapped in tantalum foil to prevent reactions with the tubing. The tubes were evacuated, backfilled with argon and subsequently sealed. Homogenization and solution heat treatments were conducted at temperatures between 1100°C and 1420°C, using a Lindberg high temperature furnace for various times up to 168 hours. Different cooling rates, furnace cooling (10°C – 20°C/min), air cooling (>100°C/min) and water quenching (>1500°C/min), were applied after solution treatments. Aging treatments were performed at temperatures between 760°C and 1100°C for various times up to 400 hours. All sample heat treatment histories started with the as-received condition, i.e. no attempts were made to re-use a sample.

3.3 Metallography

Metallographic analysis at low magnifications was conducted to reveal the microstructures after heat treatments and creep deformation. Heat treated samples for metallographic analysis were grounded with abrasive papers followed by electro-polishing at a temperature between –30°C and –35°C, using a solution of 64% methanol + 31% butanol + 5% perchloric acid. On the other hand, creep deformed samples were only mechanically polished using a Fe₂O₃ solution, in order to preserve the deformed microstructural characteristics. An etchant of 12 ml H₂O + 25 ml glycerol + 12 ml HNO₃ + 1 ml HF was used to reveal microstructural features. All samples were examined in an Olympus PMG3 optical microscope.

3.4 Electron microscopy

Scanning electron microscopic (SEM) analysis was conducted to determine the distribution of refractory alloying elements, W and Mo, using backscatter image technique, and to reveal the fracture features of the tensile specimens. Heat treated samples for SEM analysis were prepared using the same technique as for metallographic analysis, and were examined in a Hitachi S-570 and a Philips XL30S field emission gun scanning electron microscope operating at an accelerating voltage between 5 kV and 20 kV.

Image analysis of SEM backscattered electron images (BEI) was used to determine the distribution of heavy elements (W and Mo) along the cast bars owing to the difficulty in obtaining such result using SEM-EDS semi-quantitative analysis, due to the low alloying content. More than 18 SEM-BEI images were randomly taken within each end of the cast bar in order to adequately characterize the distribution of heavy elements.

Precipitates and deformation structures were analyzed using transmission electron microscopes (TEM). Samples for TEM analysis were mechanically polished to about 50 μm in thickness, followed by electrolytic jet-polishing at a temperature between -45°C and -50°C , using the same solution as for electro-polishing. TEM foils were examined using a Philips EM201 transmission electron microscope equipped with a goniometer capable of foil rotation and tilting. A Philips CM20 FEG transmission electron microscope capable of generating a fine spot size (as small as 1.2 nm) was used to determine the crystalline structure of precipitates by electron diffraction analysis. Compositions of the precipitates were determined by collecting energy dispersive x-ray spectrums (EDS) and x-ray maps in the TEM.

For each microstructural condition, multiple foils were examined to determine the precipitate particle size by measuring the maximum projected area of the particles from the TEM micrographs. Creep deformed structures were examined by preparing TEM foils from the gauge section of crept specimens so that the foil normal was parallel to the stress axis. The angle between the stress axis and the plane of the lamellar interfaces of individual lamellar grains can be determined from the goniometer tilt angle when the lamellar interfaces are tilted parallel to the incident electron beam.

3.5 X-ray diffraction analysis

X-ray analysis was used to determine the existence of β phase after heat treatments. X-ray spectrums were obtained from heat-treated samples using a Rigaku x-ray diffractometer with a Cu $K\alpha$ source ($\lambda = 1.5405$ nm, 40 kV, 30 mA) at a goniometer rotation speed of 2°/min. Identification of diffraction peaks was performed using a standard powder diffraction files (PDF) for x-ray diffraction analysis.

3.6 Microhardness test

To minimize the number of specimens for tensile and creep tests, precipitation hardening was investigated to obtain a preliminary result about the effect of precipitation on mechanical properties by analyzing the microhardness of lamellar grains. Samples for microhardness test were prepared using the same technique as for metallographic analysis, and were tested using a Leitz Miniload hardness tester with a load of 300 g. Typically, microhardness of more than 10 lamellar grains were measured for each sample in order to reflect adequately the variations in hardness measurement due to grain orientation and lamellar interface spacing effects.

3.7 Tensile and creep tests

Samples for tensile and creep tests were prepared according to ASTM standard E-8 (Fig. 3.1). The gage section of the samples was about 22 mm in length and 4 mm in diameter. All surfaces of tensile and creep samples were machined by low stress grinding.

Tensile tests were conducted using an MTS 810 Material Test System (MTS) at a strain rate of 2×10^{-3} mm/mm/s at ambient temperature and $760^{\circ}\text{C} \pm 1^{\circ}\text{C}$. The tensile strain was measured using an MTS quartz rod strain gage at both ambient and elevated temperatures.

Creep samples were tested in tension in air at $760^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and a constant load corresponding to an initial stress of 207 MPa. Creep load was applied to the sample all at once, and strain measurement began prior to loading. Creep strain was measured with an LVDT equipped extensometer attached to grooves in the specimen shoulders, providing strain resolution of at least $\pm 5 \times 10^{-4}$ mm/mm. The LVDT voltage was recorded by a PC based data acquisition system at an initial time interval of 1 second. This time interval was increased as creep proceeded. The activation energy for creep was determined at 207 MPa and temperatures between 720°C and 780°C with an interval of 20°C . Deformation structures were analyzed for samples crept for 24 hours ($<0.8\%$ strain), crept to 1.8% creep strain and crept to the onset of tertiary creep. For these interrupted tests the creep load was maintained until the sample cooled to room temperature, thereby ensuring the creep deformation structure was retained. Repeating creep tests of samples of the same condition show good reproducibility of primary creep and the minimum strain rate, with a creep strain difference of less than 10%, as shown in Fig. 3.2.

4 Microstructure of the As-received Material

The as-cast + HIPped bars of the TiAl-WMoSi alloy have a predominantly columnar grain structure in the radial direction (Fig. 4.1). Within each of the columnar grains, α_2 and γ lamellae formed as a result of the solid state phase transformation during cooling from the α single-phase region to the $\alpha_2 + \gamma$ two-phase region (Fig. 2.1). HIPping at 1260°C and 172.5 MPa for 4 hours did not alter the cast microstructure appreciably. Metallographic analysis revealed that the microstructure is not uniform along the cast bars, with more equiaxed γ and equiaxed lamellar grains present in the centre region of the top portion (Fig. 4.2), due to the lower temperature gradient within the last solidified (top) portion during casting.

The β phase, enriched in W and Mo [155,156], usually having a cuboidal shape, forms either within the cast lamellar grains (Fig. 4.3) or along the columnar grain boundaries. Image analysis of the β phase area fraction in SEM backscattered electron images (Fig. 4.4) revealed indirectly that the alloying elements distribute unevenly along the cast bars, with W and Mo segregated to the bottom portion of the cast bars, as evident by a higher density of W and Mo rich β particles in the bottom portion of the cast bars (Fig. 4.5). The segregation of W and Mo in the bottom (first solidified) portion might be associated with the effect of gravity during casting. However, the cooling rate after casting may be fast, making it unclear whether there is sufficient time for W and Mo to segregate to the bottom before solidification is complete.

SEM-EDS semi-quantitative analysis does not show any significant compositional difference of Ti and Al along the cast bar (Fig. 4.6).

Thus, the cast bar generally has a columnar lamellar structure with more equiaxed γ and equiaxed lamellar grains existing in its top section and more W and Mo segregated to the bottom section.

5 Microstructure Modification – Homogenization and Solution Heat Treatments

For turbine blade applications, cast γ based alloys are more competitive than those made by wrought or powder metallurgy methods, due to the low cost of the casting process. However, the as-cast + HIPped microstructure presented in the previous chapter would be expected to have unacceptably low ductility and strength for many applications, due to the coarse and nonuniform cast lamellar structure that is not readily removed by HIPping. Therefore, microstructural modification through heat treatment has to be applied to the as-cast lamellar structure to obtain a more uniform microstructure with improved compositional and microstructural homogeneity. A microstructure containing both cast lamellar columnar grains and some newly formed fine equiaxed γ grains, namely cast duplex structure, can be obtained after heat treating the as-cast microstructure in the $\alpha_2 + \gamma$ phase region. The TiAl-WMoSi alloy of the cast duplex condition has superior creep resistance to most other near γ -TiAl alloys, as mentioned in section 2.5. However, the duplex structure has higher tensile strength and ductility but lower creep resistance, compared to fully lamellar structure (section 2.2.1). It has been suggested that a stable fully lamellar structure with small grain size, serrated/interlocked grain boundary morphology, fine lamellar interface spacing and fine precipitation particles may have a good balance of high temperature strength, fracture toughness, creep resistance and room temperature ductility (section 2.2). Therefore, the objective of the homogenization and solution heat treatments is to modify the microstructure of the investment cast and HIPped TiAl-WMoSi alloy to obtain a uniform fully lamellar structure with a relatively small grain size and fine lamellar interface spacing, without β or other precipitate particles.

5.1 Selection of heat treatment process

As described in section 2.2.3, thermo-mechanical processing (TMP) and thermo-mechanical treatment (TMT) can produce lamellar structures with small lamellar grains [104,105]. However, components produced by investment casting cannot be subjected to either TMP or TMT due to the specific requirement of the casting geometry and therefore, only heat treatment can be applied to these components to obtain a lamellar structure. Therefore, the only option available to produce a fully lamellar structure requires a heat treatment containing at least a solution treatment at above the α transus temperature (T_α).

Solution heat treatment in the α phase region is required to produce a fully lamellar structure [7,24], however, such a heat treatment has to be performed for a short time, since long time exposure in the α phase region will lead to unwanted grain growth. For the TiAl-WMoSi alloy, heat treatment in the α phase region for a short time results in a lamellar structure with undissolved cast β particles inside the lamellar grains (Fig. 5.1), suggesting that the short time solution treatment is not sufficient to produce a β -free fully lamellar structure. Moreover, the lamellar grain size in the cast + HIPped bars varies significantly (Fig. 4.2), implying that even extended heat treatment in the α phase region is unlikely to generate a uniform lamellar structure with a relatively small grain size.

In the cast materials, homogenizing/annealing the cast lamellar structure at temperatures below the α transus temperature (T_α) for a long time followed by a solution heat treatment in the α phase field can also produce a fine-grained fully lamellar structure [7]. Thus, to obtain a fine grained fully lamellar structure with the casting Ti-Al alloys, a

process containing a homogenization heat treatment below T_α followed by a solution heat treatment above T_α should be applied. A subsequent aging heat treatment below the $\alpha \rightarrow \alpha_2 + \gamma$ eutectoid temperature (T_e) (Fig. 2.1) to stabilize the resultant lamellar structure is also necessary [24]. As such, for the cast TiAl-WMoSi alloy, a heat treatment consisting of a homogenization treatment below T_α temperature, a solution treatment above T_α temperature and an aging treatment at below T_e temperature should be applied to the as-cast and HIPped material to obtain a fine-grained fully lamellar structure, as shown schematically in Fig. 5.2.

For this heat treatment strategy, suitable temperatures and times for the homogenization, solution and aging treatments were sought, in order to obtain a fully lamellar structure with reasonably small grain size and fine dispersed precipitate particles. Also, the cooling rate after solution treatment (Fig. 5.2) had to be very well controlled to obtain a precipitate-free fully lamellar structure with fine lamellar interface spacing and interlocked/serrated grain boundaries, but avoid the formation of unwanted Widmanstätten γ (γ_w) and massively transformed γ (γ_m) structures (section 2.1.2).

5.2 Homogenization and solution heat treatments

Given the forgoing, a homogenization heat treatment at a relatively low temperature prior to solution treatment in the α phase region was applied to dissolve cast β and silicide particles. This homogenization heat treatment should proceed so that no significant grain growth occurred during heat treatment, while cast β and silicide particles are dissolved. Heat treatment at 1010°C for 50 hours resulted in the formation of some equiaxed γ grains in the cast columnar lamellar structure (Fig. 5.3), producing a duplex

structure. After this heat treatment the microstructure along the cast bar was still not uniform. This heat treatment was applied after casting by other researchers [19,20] and the resultant microstructure is generally referred to as a cast duplex structure.

Heat treatments in the $\alpha_2 + \gamma$ region were conducted in order to determine a suitable temperature-time balance for the homogenization heat treatment (Appendix 1), and the results show that heat treatment below 1140°C cannot easily eliminate the cast lamellar structure. Heat treatments at temperatures between 1160°C and 1300°C revealed many equiaxed γ grains within the cast lamellar structure, while heat treatment above 1320°C caused significant grain growth (Fig. 5.4). Therefore, homogenization heat treatments at temperatures below 1300°C can be considered.

A fully lamellar structure was obtained within the bottom portion of the cast bars, after homogenization at 1300°C for 20 hours plus solution treatment at 1360°C for 30 minutes, followed by furnace cooling (Fig. 5.5a). Preliminary homogenization heat treatment at 1180°C for 100 hours before this heat treatment assisted the formation of a fully lamellar structure with a moderately large grain size of about 600 μm (Fig. 5.6a). Extended thermal exposure at relatively low temperature, usually between 900°C and 1200°C, leads to discontinuous coarsening of α_2 lamellae, thereby producing a mixed structure of fine lamellar and equiaxed γ grains [7,106]. This mixed structure provides more nucleation sites for α grains, forming smaller grains during subsequent solution treatment. However, the solution treatment temperature has to be at least 1380°C to obtain a fully lamellar structure within the top portion of the cast bar (Figs. 5.5b and 5.5c). The difference in α transus temperatures along the cast bar is probably due to the difference in concentrations of W and Mo, i.e. addition of W and Mo might decrease α

transus temperature, since no significant compositional difference of Ti and Al was observed along the cast bar (section 4). The microstructural response of TiAl-WMoSi alloy to heat treatment is summarized in Table 5.1.

A difference in lamellar grain size after the 1380°C solution treatment can be observed across the cast bar in the top portion, with larger lamellar grains in the outer region and smaller ones in the centre region of the top portion of the cast bar (Fig. 5.6b). X-ray diffraction analysis shows no obvious evidence that β phase exists in the fully lamellar structure derived from the homogenization + solution heat treatment (Fig. 5.7). Hence, *a virtually β -free fully lamellar structure along the entire cast bar can be obtained after homogenization heat treatment at 1180°C for 100 hours and 1300°C for 20 hours, followed by solution treatment at 1380°C for 20 minutes.*

Table 5.1 Microstructural response of TiAl-WMoSi alloy to heat treatment

	top of the cast bar	bottom of the cast bar
cast + HIPped.	lamellar columnar grains with fine equiaxed γ grains at the center region.	large lamellar columnar grains.
1010°C/50hrs.	cast duplex.	cast duplex.
1300°C/20hrs.	cast duplex with γ grains coarser than above.	cast duplex with γ grains coarser than above.
1300°C/20hrs + 1360°C/30min, FC, 10°C – 20°C/min.	nearly lamellar structure with undissolved γ grains.	fully lamellar (FL) structure, average grain size about 500 μm ..
1180°C/100hrs + 1300°C/20hrs + 1360°C/30min, FC.	nearly lamellar structure with undissolved γ grains.	FL, average grain size about 600 μm .
1300°C/20hrs + 1380°C/20min, FC, 10°C – 20°C/min.	FL with few β particles, average grain size about 750 μm .	FL with some β particles, average grain size about 800 μm .

Despite the foregoing conclusion, it is very difficult to obtain a *uniform* fully lamellar structure with moderate grain size ($\leq 500 \mu\text{m}$) for the whole cast bar, due to the highly non-uniform as-cast + HIPped microstructure. Complete homogenization may be obtained after extended heat treatment at even higher temperature, however, this will

cause significant grain growth. Moreover, while homogenization at 1180°C for 100 hours is helpful in obtaining a lamellar structure with relatively small grains, it is also a time consuming process. Therefore, the homogenization and solution heat treatments for the cast material in this research are selected to be 1300°C/20 hours + 1380°C/20 minutes. The microstructure in the top portion of the cast bar after this heat treatment followed by controlled cooling is shown in Fig. 5.6b, while that in the bottom portion is quite similar but the lamellar grains at the centre of the cast bars are much coarser. The grain size of this microstructure varies from 400 – 1000 μm , giving an average grain size of about 800 μm , while some very large lamellar grains, of about 1.8 mm in size, exist at the edge of the cast bar.

5.3 Control of cooling rate after solution heat treatment

Previous work demonstrates that furnace cooling (FC) from the α phase region produces a fully lamellar structure with serrated lamellar grain boundaries, but a wide lamellar interface spacing [44]. For many γ TiAl alloys, increasing the cooling rate after solution treatment refines the lamellar interface spacing and thereby improves the creep resistance [50,51,72-75]. However, in the current alloy, air cooling (AC) after solution treatment produces a mixed structure of lamellar grains, Widmanstätten γ structure (γ_w) and massively transformed γ structure (γ_M), Fig. 5.8a, with some β particles mostly distributed along lamellar/ γ_w (Fig. 5.8b), γ_M/γ_M (Figs. 5.8b and 5.8c), and lamellar/ γ_M interfaces. β particles can also occasionally be observed inside the γ_M grains (Fig. 5.8c).

After furnace cooling from the α phase field, neither γ_w nor γ_M structures exists, but β particles can be occasionally found along the lamellar grain boundaries and at lamellar

interfaces (Fig. 5.9a), in the top portion of the cast bar. β particles can also be observed inside lamellar grains and along grain boundaries in the bottom portion of the bar (Fig. 5.9b). Silicide particles are seldom observed in either air cooled or furnace cooled samples. Therefore, neither air cooling nor furnace cooling produces the desired precipitate-free microstructure. The solution to this cooling rate problem will be presented in section 5.3.2. However, it is interesting to investigate the formation of the β phase associated with the γ_W and γ_M structures, as presented in the next section.

5.3.1 Formation of the β phase associated with the γ_W and γ_M structures

TEM-EDS results show that the solubility of W and Mo in the γ phase is lower than that in the α_2 phase, which is consistent with previous study [99]. Furthermore, W and Mo are distributed unevenly in the α_2 phase (Fig. 5.10). This compositional difference is also evident in SEM backscatter images which reveal a high contrast between α_2 and γ plates (Fig. 5.11), with α_2 plates brighter than γ plates as a result of higher W and Mo concentration in the former. The β particles seen along the lamellar/ γ_W and γ_M/γ_M boundaries (Figs. 5.8b and 5.8c) may form as a result of the rejection of W and Mo existing in the previous α phase to the interface area, during the $\alpha \rightarrow \gamma_W/\gamma_M$ transformation upon fast cooling. That is, the formation of β particles proceeds via a phase transformation of $\alpha \rightarrow \gamma_W/\gamma_M \rightarrow \gamma_W/\gamma_M + \beta$. However, the formation of these β particles has to be very fast due to the fast cooling rate, which is at least 100°C per minute during air cooling. In other words, formation of β particles within γ_W and γ_M structures is complete in 3 – 5 minutes during continuous air cooling, since such a phase

transformation is unlikely to proceed at relatively low temperatures (<800°C) during cooling, due to the low diffusion rate of W and Mo in the alloy.

The formation of β particles associated with γ_W and γ_M structures discussed above, $\alpha \rightarrow \gamma_W/\gamma_M \rightarrow \gamma_W/\gamma_M + \beta$, relies on the diffusion of W and Mo in the high temperature range. However, diffusion of W and Mo in the alloy may be quite slow due to the atomic size and mass of both elements. Another possibility is that, during air cooling from the α phase field, some of the α phase transforms to γ_W and γ_M structures, by $\alpha \rightarrow \gamma_W$ and $\alpha \rightarrow \gamma_M$, while areas rich in W and Mo (Fig. 4.4) undergo a transformation of $\alpha \rightarrow \gamma_W/\gamma_M + \beta$. Consequently, areas rich in W and Mo may act as obstacles to the movement of γ_W and γ_M grain boundaries thereby inhibiting the grain growth of these two structures. In this case no diffusion of W and/or Mo would be necessary.

In order to determine if diffusion of W and/or Mo is involved in the formation of β particles in air cooled material, one sample was heat treated at 1300°C/20hr + 1380°C/20min and quenched into water. W- and/or Mo-rich areas were not observed within the α/γ_M boundary areas or inside γ_M grains (Fig. 5.12a), suggesting that diffusion of W and Mo elements may occur during air cooling, as indicated by the presence of β particles (Fig. 5.8 and Fig. 5.12b). Thus, during continuous air cooling from α phase region, β particles form along lamellar/ γ_W and γ_M/γ_M boundaries via $\alpha \rightarrow \gamma_W/\gamma_M \rightarrow \gamma_W/\gamma_M + \beta$. It is possible that only short-range diffusion of these two elements happens in the high temperature range, between 800°C and 1380°C, during the short time available. Nonetheless, the formation of β particles has to be associated with the segregation of W and Mo, due to the low diffusivity of these two elements.

5.3.2 Cooling rate control

As indicated previously, air cooling from the α phase region is not suitable for producing a truly fully lamellar structure in the TiAl-WMoSi alloy. Furthermore, furnace cooling from the α phase region leads to the formation of lamellar structure with coarse lamellae, and precipitation of β particles in an uncontrolled manner is not desirable for creep resistance improvement [44]. To overcome these problems, a furnace cooling + air cooling technique was developed to obtain a lamellar structure with relatively finer lamellar interface spacing as well as interlocked lamellae at grain boundaries [11]. In this FC + AC technique, after solution heat treatment, the material is firstly cooled by FC to a temperature in the $\alpha + \gamma$ region to obtain an interlocked grain boundary morphology and to avoid the formation of γ_M and γ_W structures. The material is then cooled by AC to obtain a refined lamellar interface spacing and to avoid excessive precipitation [11]. In this process, the selection of the FC to AC transition temperature is critical, since too high a FC to AC temperature will result in the formation of γ_M and γ_W structures, whereas too low a FC to AC temperature leads to excessive β precipitation and coarse lamellae.

A series of heat treatments (Table 5.2) applied to the TiAl-WMoSi alloy showed that, after homogenization and solution treatment, FC to 1235°C followed by AC results in a fully lamellar (FL) structure in the bottom portion of the cast bar. However, such a γ_W/γ_M -free structure can only be obtained in the top portion of the cast bar when the material was furnace cooled to 1220°C followed by air cooling, after solution treatment. The obtained FL structure has small amount of β particles in lamellar grains (Fig. 5.13), and well interlocked lamellae in the grain boundary areas (Fig. 5.5c). Silicides were

rarely found in this structure. Lamellar structures obtained after the FC + AC two step cooling have an average lamellar interface spacing of about 100 – 140 nm, with a γ lamellae width between 40 and 500 nm. In contrast, after FC from the α phase field, the width of γ lamellae varies from 70 nm to about 1 μm , giving an average lamellar interface spacing of about 180 – 220 nm. As such, the FC + AC two step cooling after solution heat treatment in the α phase region can successfully reduce the lamellar interface spacing while preventing the formation of Widmanstätten γ (γ_w) and massively transformed γ (γ_M) structures.

Table 5.2 Microstructural response of TiAl-WMoSi alloy to cooling rate

	top of the cast bar	bottom of the cast bar
1300°C/20hrs + 1380°C/20min. FC.	FL with few β particles, average lamellar interface spacing 180 – 220 nm.	FL with some β particles along lamellar grain boundaries and inside lamellar grains.
1300°C/20hrs + 1380°C/20min. AC.	lamellar + Widmanstätten γ structure (γ_w) and massively transformed γ structure (γ_M).	lamellar + γ_w + γ_M .
1300°C/20hrs + 1380°C/20min. FC $\rightarrow$ 1250°C $\rightarrow$ AC.	lamellar + γ_w + γ_M .	lamellar + γ_w + γ_M .
1300°C/20hrs + 1380°C/20min. FC $\rightarrow$ 1235°C $\rightarrow$ AC.	lamellar + γ_w + γ_M .	FL.
1300°C/20hrs + 1380°C/20min. FC $\rightarrow$ 1220°C $\rightarrow$ AC.	FL with virtually no β particles, average lamellar interface spacing 100 – 140 nm.	FL with some β particles, average lamellar interface spacing 100 – 140 nm.
1300°C/20hrs + 1380°C/20min. FC $\rightarrow$ 1220°C $\rightarrow$ WQ.	FL.	FL.
1300°C/20hrs + 1380°C/20min. WQ.	γ_M + α_2 .	γ_M + α_2 .

The massive transformation, $\alpha \rightarrow \gamma_M$, however, occurred when the material was furnace cooled from the α phase region to 1250°C, followed by air cooling (Fig. 5.14a), but not after furnace cooling from the α phase region to 1235°C/1220°C followed by water quenching (Fig. 5.14b). This suggests that the formation of lamellar structure is complete at about 1235°C/1220°C, i.e. the final temperature for lamellar formation (γ_{Lf})

(Fig. 2.6) is about 1235°C for the bottom portion of the cast bar and is 1220°C for the top portion. However, increasing the cooling rate below the γ_{Lr} temperature leads to finer lamellar interface spacing, which is likely the result of refining of γ lamellae, by the formation of γ twin (γ_r) interfaces. That is, the reduction in lamellar interface spacing via FC + AC two step cooling is probably caused by the increase in γ/γ_r interfaces.

Therefore, after solution treatment, slow cooling to the γ_{Lr} temperature followed by fast cooling was necessary to avoid both massive transformation and excessive β precipitation at lamellar interfaces, which was shown by a previous study in Ti-Al-W system [11]. Also, the FC + AC two step cooling is helpful in producing a fully lamellar structure with fine lamellar interface spacing and well interlocked lamellae along the grain boundaries.

As such, after homogenization + solution treatment, the cast bars were furnace cooled to 1220°C followed by air cooling, in order to produce a fully lamellar structure and to prevent the formation of γ_M structure. This yields a lamellar structure (grain size of about 800 μm) with few β particles within the top portion of the cast bar (Fig. 5.13), whereas more β particles form in the bottom portion (Fig. 5.15). The existence of more β particles within the bottom portion of the cast bars can be attributed to the higher concentration of W and Mo (Figs. 4.4 and 4.5). With this compositional non-uniformity, it is very difficult to prevent the formation of both γ_M structure and β particles along an entire cast bar. Nevertheless, this two-step cooling process was used to produce the fully lamellar structure in this study because the γ_M structure reduced creep resistance [50,73].

In the optimally heat treated specimens, SEM backscatter imaging shows “clouds” of heavy elements (Fig. 5.16), suggesting that W and Mo are unevenly distributed inside

the lamellar grains after the homogenization + solution treatments. The existence of W and Mo segregation after long time heat treatment can be attributed to the low diffusivity of these two elements, especially W [36,53], since extended thermal exposure at high temperature has to be applied to eliminate segregation of W formed during casting [36].

In the top portion of the cast bar, β particles were seldom observed in the lamellar structure obtained by FC + AC two step cooling (Fig. 5.13), and only a very small amount of β particles were found in the lamellar structure after furnace cooling (Fig. 5.9a). However, the formation of β particles does occur during air cooling after solution treatment associated with the formation of γ_M and γ_W structures. That is, during cooling from the α phase region, the formation of β particles is predominantly associated with Widmanstätten and massive transformations at intermediate cooling rates, if no subsequent aging is applied. This result may seem anomalous, since it might be expected that β formation is more likely to occur at slower rates. However, the absence of β particles in the lamellar structure within the top portion of cast bar obtained by FC + AC two step cooling (Fig. 5.13) can be explained by the formation of α_2 lamellae, which has higher W and Mo solubility (Figs. 5.10 and 5.11). The small amount of β particles in the lamellar structure after furnace cooling (Fig. 5.9a) may be a result of precipitation due to the diffusion of W and Mo during continuous furnace cooling.

Silicide particles are not found in the fully lamellar structure after furnace cooling, suggesting that slow cooling from the α phase field, at a cooling rate of about $10^\circ\text{C} - 20^\circ\text{C}/\text{min}$, will not cause silicide precipitation. This indicates that the process of silicide precipitation is much slower than that for β precipitation.

The results indicate that FC + AC two step cooling can control both the lamellar interface spacing as well as subsequent formation of β particles. However, in order to prevent the formation of γ_w and γ_M structures, the required temperature at which air cooling starts for the top portion is slightly lower than that for the bottom portion of cast bar. Therefore, the lower FC to AC transition temperature (1220°C) was chosen to simplify the heat treatment applied to the cast bar and consequently, some β particles can be expected in the bottom portion due to the higher W and Mo concentration (Fig. 4.4). This suggests that the selection of FC to AC transition temperature is critical to avoid the formations of γ_w/γ_M structures and β particles, i.e. higher FC/AC transition temperature may not be able to prevent γ_w/γ_M formation, whereas lower FC/AC transition temperature may not prevent the formation of β particles.

5.4 Summary

The main objective of this part of the thesis work was to modify the microstructure of cast + HIPped TiAl-WMoSi alloy to obtain a uniform fully lamellar structure with a relatively small grain size, since it has been well documented that a fully lamellar structure with small grain size and finer interface spacing as well as interlocked grain boundaries will have a balanced set of strength, ductility, fracture toughness, and creep resistance. Solution treatment in the α phase region was applied to the as-cast and HIPped alloy in order to obtain a fully lamellar structure. The lamellar structure obtained after a short solution treatment in the α phase region has highly non-uniform lamellar grains across the cast bar, with some undissolved β particles formed during casting. A

homogenization heat treatment prior to solution heat treatment can effectively dissolve the as-cast β particles.

Air cooling from the α phase region results in the formation of a Widmanstätten γ structure (γ_w) and massively transformed γ structure (γ_M), which can be avoided by furnace cooling. However, furnace cooling leads to coarse lamellar plates and β precipitation. A two-step furnace cooling + air cooling technique was used to effectively avoid the formation of β particles and γ_w/γ_M structures and produce finer lamellar plates, as well as interlocked lamellar grain boundaries.

In the as-cast and HIPped state, both the microstructure and heavy alloying elements, W and Mo, are non-uniformly distributed along a cast bar, leading to difficulty in obtaining a uniform fully lamellar structure with fine grain size. Also, non-uniformly distributed W and Mo along the cast bar affect the temperature at which fast cooling should start: FC to a higher temperature (e.g. 1235°C) followed by AC results in γ_w/γ_M structures in the top portion of the cast bar, while FC to a lower temperature (e.g. 1220°C) followed by AC leads to β precipitation in the bottom portion. Consequently, γ_w/γ_M structures and β precipitation cannot both be avoided during the subsequent cooling after homogenization + solution heat treatment. Therefore, the microstructure along a cast bar after heat treatment will be a fully lamellar (FL) structure with some β particles in the grain boundary areas and inside the lamellar grains particularly within the bottom portion of the cast bars.

Based on the microstructural studies presented in this section, all the material presented in subsequent sections was homogenized and solution heat treated as follows:

1300°C/20hrs + 1380°C/20min, furnace cooling to 1220°C followed by air cooling, resulting in the microstructure shown in Fig. 5.5c.

6 Microstructure Modification – Precipitation Process

Following the solution treatment, aging at intermediate temperatures can produce controlled precipitate particles along the lamellar interfaces and in lamellar grain boundary regions, as described in section 2.1.3. A fully lamellar (FL) structure with fine precipitate particles along the lamellar interfaces may have enhanced creep resistance and high temperature strength [9,10-12]. However, prolonged aging may reduce the creep life of a silicon bearing γ base TiAl alloys in the cast duplex state [77]. Also, for titanium alloys containing silicon, precipitation of silicides by aging may not contribute to the creep resistance [158-160]. The purpose of the work described in this section is to investigate the precipitation process within the FL structure produced in section 5.3 and to evaluate the influence of precipitation on hardness. Samples for precipitation analysis were cut from the top section of the cast bars to avoid the β particles formed during cooling (section 5.3.2). The tensile and creep behaviour of the material in various heat treated conditions will be described in the next chapter.

6.1 Precipitation in fully lamellar structures at intermediate temperatures

The starting microstructure was a fully lamellar structure with few β particles in the grain boundaries and inside the lamellar grains, as discussed in sections 5.2 and 5.3. Precipitation treatments were conducted between 760°C and 1100°C, at intervals of 40 – 50°C. The precipitates were identified as either β or silicide particles by TEM-EDS and x-ray mapping techniques. The β particles were enriched in W and Mo, while the silicides were almost free of these two elements (Fig. 6.1). β particles have a relatively lower aluminum concentration compared to γ lamellae, whereas silicides are depleted of

aluminum. γ lamellae have relatively lower concentrations of W and Mo, since these two elements segregate to the α_2 lamellae.

Fig. 6.2 shows the x-ray maps of Mo, W and Si for material aged at 1050°C for 24 hours. Due to the overlapping of Si ($K\alpha$) and W (M) peaks, evident from Fig. 6.1, identification of silicide particles is based on the comparison of the x-ray map for W ($L\alpha$) with that for Si ($K\alpha$) and W (M). It can be concluded that particles appearing in both W ($L\alpha$), Fig. 6.2c, and Si ($K\alpha$) and W (M) maps, Fig. 6.2d, are β precipitates, while those showing up only in the Si ($K\alpha$) and W (M) map are silicides. It is notable that silicides were very small (Fig. 6.2d), compared to the β particles, although the material is aged at a relatively high temperature. It was very difficult to identify silicides in samples aged at temperatures below 850°C, using the TEM x-ray mapping, due to the small particle size.

Very fine precipitate particles, with a projected area of 0.0002 – 0.002 μm^2 , appear within the lamellar structure when aged at 760°C for 24 hours, but the number of precipitates is small (Fig. 6.3a). As the aging time and temperature increase, both the projected area and density of precipitates increase (Figs. 6.3 and 6.4). Fine dispersed precipitate particles, most of which are thin-plate-like β particles (Fig. 6.5), can be obtained after aging at temperatures below 850°C. The thickness of β precipitates increases very slowly during aging. Silicide particles are small with a projected area of 0.001 – 0.008 μm^2 after aging at temperatures up to 850°C. Most silicides have a spherical shape (Fig. 6.4c), and are located on the α_2 side of the α_2/γ interfaces and at γ/γ interfaces, but they are also found within the γ plates. In contrast, nearly all β particles are within the α_2 lamellae. Precipitation of β particles shows a strong orientation

relationship with the α_2 matrix (Figs. 6.3, 6.4a and 6.4b), and the precipitate distribution within the lamellar structure is inhomogeneous (Fig. 6.4a).

As the aging temperature further increases, the β particle size increases (Fig. 6.6) and they become farther apart, compared to that after aging at relatively lower temperatures (Figs. 6.3 and 6.4). Furthermore, at temperatures above 900°C the β particles formed have a lower aspect ratio than those developed from lower aging temperatures and they are unevenly distributed within the lamellar grains. β particles usually grow extensively within the α_2 lamellae (Fig. 6.6d), and they develop an ellipsoidal shape when aged at relatively high temperatures (Fig. 6.6c). On the other hand, the size and volume of silicides become slightly larger at higher aging temperatures. These silicide particles usually precipitate on the α_2 side of the α_2/γ and γ/γ interfaces, as reported in other silicon containing γ -TiAl alloys [9,15,16], but they have relatively smooth boundaries compared to β particles and different morphologies (Fig. 6.6c). The size of these silicides is quite small, only 0.002 – 0.02 μm^2 (projected area), after aging at 1050°C for 24 hours. The microstructural response of the FL structure to aging treatment is summarized in Appendix 2. The TEM electron diffraction pattern analysis reveals that silicide particles formed in this alloy have a hexagonal structure with $a = 0.765$ nm and $c = 0.527$ nm (Fig. 6.7), while β precipitates have an ordered BCC structure with $a = 0.3208$ nm (Fig. 6.8), close to those of Ti_5Si_3 type [16] and B2 [30] structures respectively.

From these observations, it can be concluded that at low aging temperatures (<850°C), β particles form predominantly on the α_2 side at the α_2/γ interfaces and grow in an uneven two-dimensional mode, as illustrated in Fig. 6.9. The uneven two-dimensional

character of β precipitation suggests that the growth of β particles might have an orientation relationship with α_2 matrix of $\{0001\}_{\alpha_2} // \{110\}_{\beta}$ and $\langle 11\bar{2}0 \rangle_{\alpha_2} // \langle 1\bar{1}1 \rangle_{\beta}$ [161]. The formation of β precipitates in the lamellar structure during aging is likely associated with the distribution of W and Mo in α_2 lamellae. Almost all of the β particles, enriched in W and Mo (Fig. 6.1), are within the α_2 plates when aged in the low temperature range, and grow into the γ plates only at higher aging temperatures (Fig. 6.6d).

The presence of β at temperatures between 760°C and 1260°C suggests that there is an $\alpha_2 + \gamma + \beta$ region within this temperature range for this alloy, consistent with the observations that an $\alpha_2 + \gamma + \beta$ region can exist in γ base alloys containing β stabilizing elements [34,162]. W and Mo are β stabilizing elements, and during aging at high temperatures (>900°C in this alloy) some areas within the lamellar structure enriched in these two elements probably undergo an $\alpha_2 + \gamma \rightarrow \alpha_2 + \gamma + \beta$ transformation. The formation of β precipitates proceeds at the expense of α_2 and then γ phases. At low aging temperatures, a transformation of $\alpha_2 \rightarrow \beta$ seems to be more likely for the formation of β precipitates, since almost all of the β particles precipitate on α_2 side of the α_2/γ interfaces and grow within α_2 lamellae.

Based on these microstructural observations, it is speculated that silicide precipitation strengthening of the lamellar structure is limited in the TiAl-WMoSi alloy. For 24 hour aging, the density of silicide particles is very low after aging at temperatures below 850°C. Most of the precipitates are β particles, which may have the predominant influence on strengthening the lamellar structure. Aging at temperatures above 900°C

leads to increased silicide precipitation however, and at the same time, results in considerable growth of β particles. Silicides at the α_2/γ interfaces growing into the α_2 lamellae are probably formed by the transformation $\alpha_2 \rightarrow \gamma + \text{silicides}$ [9], however, silicides at the γ/γ interfaces and within γ plates are more likely formed via a phase transformation of $\gamma \rightarrow \gamma + \text{silicides}$.

Aging at temperatures below 850°C can produce a fine dispersed precipitation of silicides and β particles (Figs. 6.3 and 6.4), which may improve creep resistance of this alloy, owing to the smaller particle size and relatively higher density of precipitation particles. It was postulated that precipitates at lamellar interfaces and/or inside lamellae might act as obstacles to dislocation movement during creep [9,12,44,157]. Aging at temperatures above 850°C may speed up precipitation processes, but it also causes unwanted growth of β particles and a reduced density of β particles (Fig. 6.6).

6.2 Kinetics of β precipitation

Precipitation of β particles is broadly reported [11,14,157] and it was suggested that blocky-shaped β resulted from segregation during solidification, whereas rod-like β formed from lamellar coarsening (Fig. 2.7) and needle-shaped β phase precipitated during annealing [14], as mentioned in section 2.1.3. However, the kinetics of β precipitation in γ base TiAl alloys has not yet been well documented. The kinetics of the formation of thin-plate shaped β precipitate particles (Fig. 6.5) in TiAl-WMoSi alloy will be analyzed in this section.

A 'short circuit-type mechanism' of mass transport was proposed [163,164] for the lengthening kinetics of plate-like θ' precipitates in an Al – 4at% Cu alloy: misfit

dislocations distributed on the broad surface of θ' plates (Fig. 6.10a), and those around the edges of precipitate plates provide suitable conduits for accelerated diffusion, leading to faster lengthening of the θ' plates than allowed by volume diffusion. On the other hand, thickening of the plate-like precipitates would be based on a ledge mechanism (Fig. 6.10b) in which ledges would spread across the broad faces of θ' plates at volume diffusion-controlled rates. Application of Fick's first law to the short circuit type diffusion controlling lengthening of plate-like precipitates yields the size of precipitates as a function of time as [164]:

$$l^2/t \propto D, \quad \text{..... (6-1)}$$

where l is the length of plate-like precipitates, and D is the solute diffusivity. If only one thermally activated diffusion process is present, the temperature dependence of diffusivity yields an Arrhenius type equation $D = D_0 e^{-Q/RT}$, with Q being the activation energy of solute diffusion. Therefore, equation (6-1) becomes:

$$l^2/t \propto D_0 e^{-Q/RT}, \quad \text{..... (6-2)}$$

Noticing that the lengthening of plate-like θ' plates in Al – 4at% Cu alloy proceeds via relatively even two-dimensional growth [163], and that in the TiAl-WMoSi alloy β particles grow in an uneven two-dimensional mode (Fig. 6.9), probably due to the orientation effect mentioned previously, we may modify equation (6-2) to

$$A/t \propto D_0 e^{-Q/RT}, \quad \text{..... (6-3)}$$

where A is the projected area of β particles (Figs. 6.3-6.6), assuming that the growth of both thin platelet- and ellipsoid-shaped β particles proceeds in a manner analogous to the 'short-circuit mechanism'. Thus, we may be able to determine the β precipitation kinetics by measuring the maximum projected area of β particles after various aging

treatments. During aging between 760°C and 950°C, the projected area (A) of β particles increases linearly as aging time increases (Fig. 6.11), indicating that after nucleation, β particles grow at a nearly constant rate. As the aging temperature increases, the growth rate of β particles increases up to 850°C. However, an increase in the growth rate is not observed at a higher aging temperature of 950°C.

The Arrhenius plot for the growth rates (A/t) of β particles is shown in Fig. 6.12. Noticing that the growth rate of β particles increases as aging temperature increases only up to 850°C, it may be assumed that there may be a change in growth mechanism of β particles from low aging temperatures to higher ones, showing a possible breakdown of the 'short circuit-type mechanism'. Bearing this in mind, an activation energy of about 366 kJ/mol is appropriate for β growth at temperatures between 760°C and 850°C. It has been shown that for self-diffusion of major elements in titanium aluminides, $Q_{Ti} = 250 - 291$ kJ/mol, $Q_{Al} = 360$ kJ/mol in γ -TiAl [149,150], while $Q_{Ti} = 288$ kJ/mol, $Q_{Al} = 395$ kJ/mol in Ti_3Al [165,166]. Also, the activation energy for substitutional diffusion of Nb is 339 kJ/mol in Ti_3Al [166], where Nb is a substitutional element in the alloy. Considering the similar electronic configurations among Nb, W and Mo atoms (group V for Nb, group VI for W and Mo), as well as the atomic mass of each element, the diffusivity of Mo is likely close to that of Nb, and that of W is lower than that of Nb. Therefore, activation energies for substitutional diffusion of W and Mo should be higher than that for Nb, that is, $Q_W \geq Q_{Mo} \geq 339$ kJ/mol, leading to the low diffusivity of W [36,53]. The activation energy derived for β formation is 366 kJ/mol at temperatures between 760°C and 850°C, which is greater than that for substitutional diffusion of Nb [166]. In addition, β precipitates are enriched in W and Mo. This suggests that the

growth of β particles at between 760°C and 850°C could be controlled by the diffusion of W and Mo.

It is difficult to determine quantitatively if the growth of β particles during aging is controlled by substitutional diffusion of W and Mo, since activation energies for substitutional diffusion of W and Mo in γ -TiAl are not available. However, it is more likely that the diffusion of W and Mo at β/α_2 and β/γ interfaces accounts for the growth of β particles at temperatures between 760°C and 850°C. This is based on the constrained growth of β particles at the interfaces between α_2 and γ plates and the two-dimensional growth of β platelets (Fig. 6.9) between 760°C and 850°C. The two-dimensional growth of β platelets suggests that diffusion of W and Mo across the β/γ interface and the β/α_2 interface that is parallel to the β/γ interface is very slow, compared to the interfacial diffusion along the β/α_2 and β/γ interfaces, which is similar to the situation described by the 'short-circuit mechanism' [163,164]. The limited diffusion of W across the β/γ interfaces in a Ti-Al-W alloy was recently reported [167]. Diffusion of W and Mo at β/α_2 and β/γ interfaces is likely easier than that within the α_2 and γ phases, due to the misfit dislocations at these semi-coherent interfaces [54].

The growth rate of β particles at 950°C does not agree with the derived Arrhenius relationship for aging between 760°C and 850°C, suggesting that the growth process of β particles at temperatures above 850°C changes. Consistent with this result, a change in the morphologies of β particles (from thin platelets to ellipsoids) occurs at about 900°C (Fig. 6.6), which may also imply that the mechanism for β growth changes. It is also notable that the distribution of β particles formed at low aging temperatures, below

850°C. is relatively uniform (Figs. 6.3 and 6.4), compared to that at high aging temperatures (Fig. 6.6). The change from β formation with a strong orientation (Fig. 6.4b) at low aging temperatures to less orientation dependence (Fig. 6.6) at higher aging temperatures suggests a loss of coherency between β particles and the matrix, which could be explained by a decrease in the surface area to reduce energy at high aging temperatures. Such growth has to be accompanied by greater diffusion distances of the major elements existing in the particles, i.e. W and Mo in this alloy, along the β/α_2 and β/γ interfaces. However, the comparatively low growth rate at 950°C does not agree with this mechanism that requires W and Mo interfacial diffusion. Also, the more inhomogeneous distribution of β particles formed at relatively higher temperatures (Fig. 6.6) suggests that the formation of β particles above 900°C is likely associated with the non-uniform distribution of W and Mo in the FL structure (Fig. 5.16).

It can be expected that during aging, β particles nucleate and grow via $\alpha_2 \rightarrow \beta$ transformation, as a result of diffusion of W and Mo, due to the existence of the $\alpha_2 + \gamma + \beta$ (B2) phase region at higher temperatures [34,162]. Segregation of W and Mo can be expected after casting [36,88], and complete homogenization of W is very difficult [36]. At low aging temperatures, the driving force for $\alpha_2 \rightarrow \beta$ transformation may be quite small and at the same time, the diffusivity of W and Mo is low. Consequently extensive interfacial diffusion of W and Mo along a fast path occurs in the areas rich in these two elements within α_2 lamellae, forming relatively homogeneously distributed β precipitates mostly inside the α_2 lamellae. The misfit dislocations along the β/α_2 and β/γ interfaces may serve as diffusion short circuits [164] allowing W and Mo to diffuse faster.

However, at high aging temperatures, the driving force for $\alpha_2 \rightarrow \beta$ transformation may be quite large and the diffusivity of W and Mo is higher. Two possible processes may be associated with the growth of β particles during aging at higher temperatures. One possibility is that during the early stage of aging, areas rich in W and Mo transform to β particles rapidly due to the heterogeneous nucleation and short-range diffusion of W and Mo within these areas, before extensive diffusion of these two elements can occur, as a result of increased aging temperature. As aging further proceeds, diffusion of W and Mo from surrounding areas to β particles becomes important after the $\alpha_2 \rightarrow \beta$ transformation in the W- and Mo-rich regions, leading to a reduced growth rate in the extended thermal exposure at high aging temperatures (Fig. 6.11). Thus, the initial rapid increase in projected area in Fig. 6.11 at 950°C is probably due to the $\alpha_2 \rightarrow \beta$ transformation controlled by short-range diffusion of W and Mo within the W- and Mo-rich areas, while the growth rate during subsequent aging is controlled by long range W and Mo diffusion from the surrounding areas.

The other possibility for β particle growth at higher aging temperatures is that the β particles also have a thin-plate shape and grow in an uneven two dimensional mode at the very beginning of aging. However, the time for uneven two-dimensional growth of β particles at higher aging temperatures is very short, due to the higher diffusivity of W and Mo and the loss of coherency with the α_2 matrix, compared to at low aging temperatures. The reduced growth rate during extended aging is controlled by the substitutional diffusion of W and Mo. Similarly, it can be expected that aging at relatively low temperatures for sufficient time may also result in the loss of coherency between β particles and α_2 matrix and therefore, a reduced growth rate during extremely extended

aging controlled by the substitutional diffusion of W and Mo. Thus, an activation energy not less than 370 kJ/mol can be expected for the β particle growth for extended aging, since it was suggested that 'short circuit' type diffusion have lower activation energy compared to volume diffusion [164]. Investigation of β particle size after short-time aging at high temperatures (900°C and 950°C) and that after long-time aging at low temperatures (800°C and 850°C) will be necessary to obtain better understanding of the precipitation process of β particles.

6.3 Precipitation hardening of nearly lamellar structure

As mentioned previously, precipitation in γ -TiAl strengthens this type of material. A positive effect of precipitation on creep resistance was reported in γ base TiAl alloys containing silicon and carbon [9,12,96] however, long time thermal exposure at intermediate temperatures may reduce the creep life of silicon containing γ TiAl [77]. Precipitation hardening by silicides was observed in commercial titanium alloys [168,169]. However, the silicides formed at relatively higher temperatures during aging do not strengthen the material [168]. Hardness reflects a material's resistance to plastic deformation under compression stress, which gives a preliminary indication about a material's strength since in many alloy systems, material having higher hardness usually has higher yield strength. Thus, microhardness of the FL structures of various heat treating conditions was measured to rapidly determine the resistance to plastic deformation of the TiAl-WMoSi alloy with respect to β precipitation. A load of 300 g was applied and the indentation size varies from 30 to 50 μm , much smaller than lamellar grain size (>400 μm). Microhardness measurements were conducted in such a way that

all indentations were at least 100 μm from the lamellar grain boundaries so that hardness arising from lamellar grain boundaries was avoided.

The increase in room temperature hardness of the lamellar grains is not appreciable for the specimen aged at 800°C for 24 hours (Fig. 6.13) whereas the hardness increases by about 10 to 15% after aging at 850°C for 24 hours, compared to the unaged material. After aging for 24 hours, the hardness versus temperature curve shows a hardness peak at 850°C, and the hardness of lamellar grains drops when the aging temperature exceeds 850°C. However, a slight increase in hardness can be noticed after aging for 24 hours at 1000°C and 1050°C. When aged at 800°C, hardness increases slowly as the exposure time increases, but not by more than 10% even after aging for over 200 hours (Fig. 6.14). Aging above 850°C causes the hardness to initially increase significantly but then drops as the exposure time increases, exhibiting a hardness peak at about 24 hours/850°C and at about 12 hours/950°C (Fig. 6.15). The variations in hardness for each condition (Figs. 6.13 to 6.15) probably arises from the orientation differences in the lamellar grains and the inhomogeneous distribution of precipitates.

It is notable that the lamellar structure obtained via FC + AC two step cooling is harder than that via furnace cooling after solution treatment (Fig. 6.13), and this can be attributed to the reduced lamellar interface spacing after FC + AC two step cooling, as mentioned in section 5.3.2. Furnace cooling to γ_{L} temperature followed by air cooling is advantageous to obtain a fully lamellar structure with increased strength compared to one-step furnace cooling.

The maximum hardness occurs at 850°C, when the lamellar structure is aged for 24 hours, suggesting that the optimum precipitation hardening appears at a temperature

between 800°C and 900°C, which is consistent with the observations of the fine dispersed precipitate particles occurring in this temperature region (Fig. 6.4). At temperatures higher than 900°C, precipitation hardening is reduced (Fig. 6.13), which is probably due to the excessive growth of β particles. The slight increase in hardness after aging for 24 hours at 1000°C and 1050°C is probably due to the increased silicide precipitation (Fig. 6.6). Precipitation hardening after aging at temperatures above 1000°C may be of less importance, since large β particles will reduce creep resistance and degrade lower temperature ductility. Aging at 800°C for over 200 hours does not enhance precipitation hardening (Fig. 6.14), compared to aging at 850°C for 24 hours (Fig. 6.15), and therefore, aging at 800°C is less suitable for this alloy since it requires longer time to achieve the expected level of precipitation hardening. Long time exposure above 850°C leads to softening of the material (Fig. 6.15). Hence, optimum precipitation hardening can be expected after aging at temperatures between 850°C and 900°C for a reasonably short time.

6.4 Summary

Investigation of the precipitation processes in Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si reveals that aging at intermediate temperatures ranging from 760°C to 1100°C produces both β (ordered BCC structure) and silicide (hexagonal) particles, with the majority of precipitates being β particles. Silicide precipitation increases as temperature increases. However, the growth of silicides is very slow, which suggests that silicide precipitation in fully lamellar TiAl-WMoSi alloy is less important than β precipitation for strengthening the material. Therefore the major concern for the precipitation process

should be focused on the formation of β particles. β particles formed at temperatures below 850°C exist predominantly in the form of thin platelets, while those formed at higher temperatures appear as ellipsoid blocks.

Finely dispersed β precipitates can be obtained in the fully lamellar structure at temperatures below 850°C. However, extensive thermal exposure will be required at temperatures below 800°C. Meanwhile, precipitation hardening at temperatures below 800°C appears to be very slow as aging time increases. At temperatures above 900°C, the β particle size increases rapidly as temperature and time increase, and they are usually unevenly distributed inside the lamellar grains, compared to those formed at temperatures below 850°C. Overaging occurs when extended thermal exposure is applied to the lamellar structure, especially at higher aging temperatures, showing an optimum precipitation hardening at between 850°C and 900°C for about 24 hours.

Preliminary results on the kinetics of β growth give an activation energy of about 366 kJ/mol at temperatures below 850°C. This implies that at low aging temperatures the growth of β particles undergoes an $\alpha_2 \rightarrow \beta$ precipitation process controlled by diffusion of W and Mo within the β/α_2 and β/γ interface areas. At temperatures above 900°C, β particles grow very fast at the very beginning of aging, probably due to the 'short circuit' type diffusion, or the heterogeneous β formation within the areas rich in W and Mo controlled by short-range diffusion. Consequently the formed β particles grow at a much lower rate (than would be predicted using the determined 366 kJ/mol activation energy), as a result of the loss of coherency between the β particles and α_2 matrix, due to the low substitutional diffusivity of W and Mo from the surrounding areas.

7 Mechanical Property Analysis

Chapters 5 and 6 show how to obtain a fully lamellar (FL) structure, as well as precipitate particles within the lamellar grains. In this chapter the effects of precipitation on the tensile and creep properties of the fully lamellar structure are presented. Also, for comparison, tensile and creep properties of the cast duplex structure (Fig. 5.3) are also evaluated. Based on the morphology of precipitates (Figs. 6.3 through 6.6), as well as the precipitation hardening behaviour, the FL structures in the unaged, 850°C/24hr and 950°C/24hr aged conditions were selected to investigate the influence of β particles on the tensile and creep behaviour in the FL structure.

7.1 Tensile behaviour of FL structures at ambient and elevated temperatures

Tensile tests were conducted at a constant strain rate of 2×10^{-3} mm/mm/s at both ambient and elevated temperatures. It can be seen that a decrease in both tensile strength and elongation occurs at room temperature, shown in Fig. 7.1, when the cast duplex structure is eliminated and a fully lamellar structure is formed. The tensile properties are listed in Table 7.1. The lower yield strength (0.1% offset strength $\sigma_{0.1}$) of the FL structure, compared to that of cast duplex structure, can be associated with the large grain size of the FL structure [8,57]. Moreover, FL structures have lower elastic strength (σ_e or $\sigma_{0.2}$) and higher work-hardening rates than the cast duplex microstructure (Fig. 7.1), as a result of highly anisotropic and nonuniform plastic deformation in FL structures [57]. Similar results were obtained in some other TiAl alloys [8,56,57]. Furthermore, FL structures after aging especially at higher aging temperature exhibit higher yield strength ($\sigma_{0.1}$) and ultimate tensile strength (UTS), which can be attributed to the precipitation of β

particles. However, this result contradicts the microhardness measurements and will be discussed later.

Table 7.1 Tensile properties of TiAl-WMoSi alloy at ambient and elevated temperatures

	25°C			760°C		
	$\sigma_{0.1}$ (MPa)	UTS (MPa)	δ (%)	$\sigma_{0.1}$ (MPa)	UTS (MPa)	δ (%)
cast duplex.	545	583-619	0.22-0.48	391-419	485-542	0.55-0.94
FL, unaged.	320	374-387	0.22	190-220	331-372	0.5-0.53
FL, 850°C/24hrs.	335-350	429	0.23-0.24	245	358-381	0.38-0.51
FL, 950°C/24hrs.	408-422	455-477	0.17-0.19	276-315	328-414	0.13-0.49

A similar trend of β precipitation improving tensile strength is also observed at 760°C (Fig. 7.2a). It is notable that the strain of FL structures is nearly the same at low stresses (<130 MPa), regardless of the β precipitation (Fig. 7.2b). However, as the stress increases, the FL structure aged at higher temperature (950°C) is more resistant to deformation than the unaged FL structure. For all the tested structures, a decrease of about 10% in tensile strength is obtained at elevated temperature.

Fractography indicates that at ambient temperature the cast duplex structure and FL structures have similar fracture mechanisms: all the samples failed with translamellar and delamination fracture (Figs. 7.3 and 7.4). The differences in these samples are the fraction of translamellar feature and the dimension of the fracture facets as a result of cleavage along lamellar interfaces (delamination) within the lamellar regions. In the cast duplex structure (Fig. 7.3), the fracture surface has more translamellar features and the fracture facets are quite small. In the FL structures, lamellar delaminations appear frequently (Fig. 7.4) regardless of whether the FL structure is aged or not. Much larger fracture facets compared to those in the cast duplex structure (Fig. 7.3b), can be found within the lamellar grains with clear cleavage characteristics along surfaces, as a result of

the larger lamellar grain size of the FL structures. At elevated temperature, no significant change in fracture modes is observed in either the cast duplex or FL structures (Figs. 7.5 and 7.6). Translamellar and delamination fracture dominates within the lamellar regions.

The FL structures have relatively low tensile strength and ductility at both ambient and elevated temperatures (Figs. 7.1 and 7.2), compared to the cast duplex structure. This is associated with the absence of fine equiaxed γ grains within lamellar grain boundary areas as well as the larger lamellar grains of FL structures (Figs. 5.3 and 5.5c). Similar results were also obtained by other researchers [56] (section 2.2.1). Lamellar grains failed predominantly via cleavage along lamellar interfaces (Figs. 7.4 and 7.6). The larger lamellar grains in the FL structures provide an easier path for crack propagation along the lamellar interfaces, leading to lower tensile elongation.

The precipitation of the β particles may be responsible for the increased tensile strength of the aged FL structures, and higher aging temperature leads to an increased β particle size therefore the increased tensile strength (Fig. 7.1 and Fig. 7.2a). Presumably, precipitation particles formed during aging will act as obstacles to dislocation movement, making the material more resistant to plastic deformation thereby enhancing the strength. Thus, β particles formed at lamellar interfaces may strengthen the FL structures by pinning dislocation segments. It is likely that aging at 850°C for 24 hours cannot produce β particles that are as effective at hindering dislocation motion as at 950°C, so that the tensile strength is lower.

As mentioned in section 6.1, aging above 900°C leads to considerable β particle growth, and increased silicide precipitation as well. The increased silicide precipitation may contribute to the enhanced tensile strength of FL structures. Therefore, it can be

suggested that the tensile strength after aging is due to the increased β particle size and silicide precipitation leading to more effective interactions between these particles and dislocations.

The appearance of β phase in γ - TiAl is not likely to increase the ductility at ambient temperature, since β particles may act as sources of micro-cracking [41] thereby reducing room temperature ductility [81]. At elevated temperatures, the presence of large β blocks, primarily along grain boundaries, will enhance the ductility of TiAl if the temperature is high enough and/or if the strain rate is low enough [80,82,83]. However, in the current work, the formation of β particles does not have a significant influence on the tensile elongation (Fig. 7.2a), probably due to the small particle size. Moreover, the presence of β precipitates increases the resistance to deformation when the tensile stress exceeds 130 MPa (Fig. 7.2b), which can be attributed to the interactions between these precipitates and dislocations.

The FL structure aged at a higher temperature (950°C) has an increased tensile strength (Fig. 7.1) while it has a decreased microhardness (Fig. 6.13), compared to the FL structure aged at a relatively lower temperature (850°C), which may be due to the properties of material at different scales. As mentioned in section 6.3, the size of microhardness indentations is about 30 – 50 μm and all indentations were made within the lamellar grains; thus the microhardness (strength) arising from lamellar grain boundaries is neglected by microhardness measurement. On the other hand, tensile strength represents an average property of a few lamellar grains and therefore, strengthening of finer precipitates formed at lower aging temperature may be smaller, compared to the effect arising from the grain boundaries, due to the small particle size.

Aging at lower temperatures produces finer and more dispersed β particles therefore the FL structure has a higher microhardness, while aging at higher temperatures results in increased particle size but also an increased spacing between the particles therefore the FL structure a lower microhardness. However, aging at higher temperatures produces β particles that are large enough to prevent dislocations from moving, leading to the increased tensile strength.

7.2 Creep behaviour of FL structures

It can be noticed that the FL structures have decreased tensile strength at both ambient and elevated temperatures, and that aging at a relatively higher temperature leads to increased tensile strength. This section deals with the impact of FL structures on the creep behaviour. Creep tests were conducted in air at 207 MPa and 760°C to assess the creep behaviour of the fully lamellar structures. The apparent activation energy for creep was determined at 207 MPa between 720°C and 780°C.

The creep results indicate that FL structures exhibit lower steady-state or minimum creep strain rates and extended creep life, compared to the cast duplex structure (Fig. 7.7), suggesting that the FL structure is more resistant to creep than the cast duplex structure. Moreover, FL structures exhibit an extended period of secondary (steady-state) creep, compared to the cast duplex structure. Similar results have been documented by other researchers [56,63-71]. The variation of creep behaviour for each condition can be attributed to the non-uniform characteristics of casting material, particularly the grain size and orientation of lamellar grains, as mentioned in chapters 4 – 6.

For the FL structures, aging at intermediate temperatures results in a decrease in creep life, and the FL structure aged at higher temperature has shorter creep life, similar to a γ -TiAl alloy containing W and Si. This shows that aging at intermediate temperatures has a negative effect on creep life [77]. After aging, FL structures generally have a shorter secondary creep region (Fig. 7.7).

The FL structure aged at 950°C exhibits a lower instantaneous creep strain and a longer time to 0.5% creep strain during the primary creep (Table 7.2), while the unaged FL structure shows a higher instantaneous strain and a shorter time to 0.5% creep strain. This demonstrates that β particles may be beneficial for the primary creep resistance of the FL structure. It can be expected that β particles within the FL structures interact with dislocations by inhibiting dislocation segment movement during primary creep, resulting in enhanced primary creep resistance. The difference in instantaneous strain between the unaged FL structure and that aged for 24 hours at 850°C is small. However, the 850°C/24hr aged FL structure has increased time to 0.5% creep strain (Fig. 7.8). Aging at temperatures between 850°C and 950°C does not have a significant influence on the secondary creep rate of the FL structures (Table 7.2).

Table 7.2 Creep properties of TiAl-WMoSi alloy at 760°C and 207 MPa

	instantaneous strain (%)	time to 0.5% strain (hr)	steady-state creep rate (mm/mm/hr)	creep life (hr)	fracture strain (%)
cast duplex.	0.26	5	1.47×10^{-4}	405	16.09
FL, unaged.	~ 0.27	~ 3.7	$(0.9 - 1.2) \times 10^{-5}$	2759	~ 5.29
FL, 850°C/24hr.	~ 0.26	~ 18	$(1.0 - 1.2) \times 10^{-5}$	1800 +	~ 4.85
FL, 950°C/24hr.	~ 0.19	~ 38	$(1.4 - 1.6) \times 10^{-5}$	1215, 2166	4.63, 8

After aging at 950°C for 24 hours, the FL structure has the lowest instantaneous creep strain, whereas the unaged FL structure and the one aged at 850°C for 24 hours

have higher instantaneous creep strain (Fig. 7.8a). After 5 minutes of creep loading, the creep strain of the FL structures follows the sequence of unaged, 850°C/24hr aged and 950°C/24hr aged, with the unaged FL structure having the highest creep strain. This is consistent with the trend observed at 207 MPa of the tensile tests at 760°C (Fig. 7.2b). This suggests that the instantaneous creep strain of FL structures may be related to the tensile behaviour of these structures at the same temperature. At the onset of creep the structure that is more resistant to tensile deformation, i.e. higher Young's modulus and/or higher yield strength, will have a lower instantaneous creep strain upon creep loading.

The unaged FL structure, on the other hand, has a similar instantaneous creep strain and time to 0.5% creep strain as the cast duplex structure (Table 7.2). Therefore, the FL structures have longer creep life but only the aged FL structures are more resistant to primary creep than the cast duplex microstructure. However, with tensile loading to 207 MPa, the cast duplex structure has a small strain than the unaged FL structure, but with aging, the FL and duplex microstructures have similar strains in Fig. 7.2. In creep, however, the instantaneous strain in duplex microstructure is much higher (Table 2). This difference may be related to the fact that loading history is different. Investigation of tensile behaviour at 760°C and a higher strain rate may be helpful to understand the relationship between instantaneous creep strain and tensile behaviour, since it takes about 40 – 80 seconds to reach 207 MPa in tensile tests (Fig. 7.2b) but about 3 – 6 seconds in creep tests (Fig. 7.8a). Moreover, the extended creep life as well as the lower creep strain of the FL structures, compared to the cast duplex structure, can be attributed to the decreased volume of equiaxed γ grains along lamellar grain boundaries, since the γ phase

is more deformable than the α_2 phase, as described in section 2.3. The cause of decreased creep life of aged FL structures (Fig. 7.7) will be discussed later in this chapter.

In the unaged FL structure, creep fracture occurs predominantly along lamellar grain boundaries and occasionally along lamellar interfaces (Fig. 7.9). Void/cavity formation can frequently be observed in areas close to fracture surface, while fewer voids/cavities can be found in areas far from the fracture (Fig. 7.10). This suggests that the formation of voids/cavities along lamellar grain boundaries [170,171], together with subsequent intergranular crack propagation [35,131] via coalescence of voids/cavities, are the main causes of creep fracture. Interrupted creep tests show that the void/cavity formation starts during secondary creep (Fig. 7.11a) and that lamellar delamination appears during tertiary creep (Fig. 7.11b).

Crack propagation along lamellar grain boundaries via void/cavity formation also plays a very important role in creep deformation of FL structures after aging. Fewer voids/cavities can be observed in the FL structure aged at 850°C for 24 hours when crept to the secondary stage, a creep strain of about 1.8%, and voids/cavities aggregate during the tertiary creep. After fracture, creep samples of aged FL structures have features similar to those in unaged FL structure (Figs. 7.9 through 7.11). Moreover, lamellar delamination in the lamellar grains with aged FL structures becomes substantial during creep (Fig. 7.12). The increased lamellar delamination in aged FL structures suggests cracks are easier to initiate and propagate along lamellar interfaces in aged FL structures, compared to those in unaged FL microstructure.

Another interesting feature in creep deformation is the shear deformation across the lamellar interfaces. It can be clearly seen that the shear across lamellar interfaces appears

during the tertiary creep in the unaged FL structure (Fig. 7.13) while it occurs during the secondary creep in the FL structure after aging at 850°C for 24 hours (Fig. 7.14). This suggests that some change(s) of microstructure may happen during creep, and that shear across lamellar interfaces, as a result of microstructural change(s). It is likely that aging makes the lamellar structure less stable during the creep thus the early appearance of shear across lamellar interfaces, which will be further discussed in a later section.

7.3 Creep deformation structures

Several creep samples with FL structures were interrupted at primary, secondary or tertiary stages (Appendix 3), in order to examine the mechanism of creep of the FL structures. This section focuses on the deformation structures in the FL structures since deformation structures of cast duplex structure have been well documented by other researchers [156,157]. The unaged FL structure and FL structure aged for 24 hours at 850°C were chosen for this investigation. Lamellar grains with γ/α_2 interfaces nearly perpendicular or parallel to the stress direction are referred to as “hard-oriented” grains, whereas those inclined from stress direction with an angle of 20 – 70° as “soft-oriented” grains, consistent with section 2.2.1.

7.3.1 Deformation of unaged FL structure

In the unaged FL structure, some dislocations exist in the undeformed condition (Fig. 5.13), while no significant increase in dislocations can be observed in both “soft” and “hard” oriented lamellar grains (Fig. 7.15) during the primary creep. Meanwhile, lamellar grains with a “soft” orientation do not have noticeably higher dislocation activities than the “hard” oriented grains. γ/α_2 interfaces act as obstacles to dislocation

movement, as indicated by bowed dislocations between adjacent lamellar interfaces (Fig. 7.15). Also, γ/α_2 interfaces act as dislocation sources, as evidenced by dislocation loops coming out from them (Fig. 7.15b), but the number of newly formed dislocations is very limited. Formation of β particles occurred within the gauge section of unaged FL creep sample, and the size of these particles is nearly the same as in the FL structure aged at 760°C for 24 hours (Fig. 6.3a).

The density of dislocations increases (Fig. 7.16a) as creep strain increases. However, the number of dislocations within the lamellar grains is still small during secondary creep. Interactions between β particles and dislocations are occasionally observed at lamellar interfaces as indicated by arrows in Fig. 7.16b. A higher dislocation density exists within wide γ lamellae (Fig. 7.17a), in equiaxed γ grains along lamellar grain boundaries and in γ lamellae close to equiaxed γ grains (Fig. 7.17b).

The number of dislocations increases as creep enters the tertiary stage, with more dislocations appearing in the “soft” oriented lamellar grains (Fig. 7.18a), compared to those in the “hard” oriented ones (Fig. 7.18b), consistent with previous observation, as described in section 2.3.2 [125]. New dislocations emanate from interfaces during tertiary creep (Fig. 7.18a). Interactions between dislocations and β particles can be observed in both lamellar grains (Fig. 7.19) and equiaxed γ grains along lamellar grain boundaries (Fig. 7.20a). Twinning can be found within equiaxed γ grains along lamellar grain boundaries (Fig. 7.20b). α_2 decomposition occurs during tertiary creep, leaving β particles isolated in the γ matrix (Fig. 7.19b). Creep continues to be dominated by the generation and movement of dislocations until fracture occurs (Fig. 7.21). Deformation

twinning is not observed within the lamellar structure. Moreover, no trace of dynamic recrystallization of γ grains can be found.

7.3.2 Deformation of aged FL structures

Similar to the unaged FL structure, dislocations are generated during primary creep in both “soft” and “hard” oriented lamellar grains in the 850°C/24hr aged FL structure (Fig. 7.22). As the creep strain increases, the number of dislocations increases more slowly in “hard” oriented lamellar grains (Fig. 7.23a), compared to that in “soft” ones (Fig. 7.23b), during secondary creep. Interaction between β particles and dislocations at lamellar interfaces becomes evident during secondary creep (Fig. 7.24) and, at the same time, α_2 plates start to decompose leaving β particles isolated in the γ matrix (Fig. 7.24b). Dislocation activity is still quite low during secondary creep. As deformation continues, dislocation activity increases in both “soft” and “hard” oriented lamellar grains (Fig. 7.25a and 7.25b), while equiaxed γ grains along lamellar grain boundaries reveal much more dislocation activity (Fig. 7.25c). Deformation twinning can hardly be seen in lamellar grains until fracture occurs (Fig. 7.26), while new dislocations continue to be generated at lamellar interfaces during the creep (Figs. 7.23b and 7.26b).

Lower dislocation activity is observed in 950°C/24hr aged FL structure during primary creep (crept for 24 hours), with little difference in the density of dislocations between “hard” and “soft” lamellar grains (Fig. 7.27). As creep strain increases, a higher density of dislocations appears in the lamellar grains with “soft” orientation (Fig. 7.28), with dislocations interacting with β particles and lamellar interfaces. “Hard” oriented grains have a much lower dislocation density, and more dislocations appear in γ lamellae

close to equiaxed γ grains along lamellar grain boundaries (Fig. 7.29). Deformation twinning can only be found in equiaxed γ grains along lamellar grain boundaries and dynamic recrystallization of γ grains does not occur.

7.3.3 Discussion of creep deformation in FL structures

It may be concluded that creep deformation of FL structures proceeds by the deformation of lamellar grains via dislocation generation and movement, as well as the deformation of equiaxed γ grains along lamellar grain boundaries via dislocation movement and twinning, similar to the mechanisms mentioned in section 2.3.2.

It is possible that when stress is applied, both lamellar grains and equiaxed γ grains start to deform by the generation and movement of dislocations. At the very beginning of the creep, lamellar grains deform predominantly via the movement of the dislocations that already exist within the lamellar structure and therefore, both “hard” and “soft” lamellar grains reveal a similar amount of dislocations. This is shown by the unchanged dislocation density during primary creep (Fig. 7.15), compared to the undeformed condition (Fig. 5.13a). γ/α_2 interfaces act as dislocation sources (Figs. 7.15b, 7.22b and 7.27b), and they are also obstacles to dislocation movement as indicated by bowed dislocations between adjacent lamellar interfaces (Figs. 7.15, 7.22 and 7.27), as presented by other researchers [72,125,127,129].

As creep strain increases, dislocation activity increases in lamellar grains especially in those with “soft” orientations (Figs. 7.18, 7.23, 7.25, and 7.26) [125], suggesting that “soft” lamellar grains are more deformable than “hard” ones when creep strain exceeds a certain level. In other words, lamellar grains with “soft” orientation contribute more

towards overall creep strain at high strains. Wider γ plates and equiaxed γ grains provide less of an obstacle to dislocation movement, compared to narrow lamellar plates, and therefore contribute more to deformation (Figs. 7.17, 7.20 and 7.25). Interactions between dislocations and β particles (Figs. 7.16b, 7.20a, 7.24, 7.28 and 7.29a) imply that these particles are also obstacles to dislocation movement by pinning dislocation segments [127]. The process of dislocation generation and movement continues in both lamellar grains and equiaxed γ grains along lamellar grain boundaries, producing and increasing stress concentrations at boundaries [57] between lamellar grains of different orientations and at those between lamellar and equiaxed γ grains, due to the difference in deformability among constituents. Localized stress accumulation between these constituents results in void/cavity formation (Fig. 7.11a) at lamellar/lamellar and lamellar/equiaxed γ grain boundaries [170]. As creep strain further increases and enters the tertiary stage, void/cavity formation [170,171] continues and voids/cavities along the lamellar grain boundaries join together forming bigger cracks (Fig. 7.9) consequently leading to fracture.

The α_2 plates, on the other hand, begin to decompose during the secondary creep in the 850°C/24hr aged FL structure (Fig. 7.24b), while the decomposition of α_2 plates in the unaged FL structure does not occur until tertiary creep (Fig. 7.19b). This suggests that aging makes the α_2 plates less stable during the creep, which is consistent with the previous observation of an earlier shear deformation across lamellar plates in the aged sample (Figs. 7.13 and 7.14). Hence, the early decomposition of α_2 lamellae during creep is likely the major cause for the decreased creep life of aged FL structures. It is likely that the removal of W and Mo from the α_2 into the β particles [167], as a result of β

formation, makes the FL structure less stable during the creep. This leads to earlier decomposition of α_2 lamellae, since W in solution stabilizes the α_2 lamellae against dissolution and coarsening [99]. Early decomposition of α_2 lamellae decreases the volume of obstacles to dislocation movement thereby reduces the creep life of aged FL structures.

The FL structure aged at 950°C has increased primary creep resistance (Fig. 7.8) as a result of enhanced β precipitation, compared to that aged at lower temperature, which is consistent with tensile result (Fig. 7.2b). Interactions between β particles and dislocations seem to be obvious during primary creep in the 950°C/24hr aged FL structure (Fig. 7.27b), compared to those in FL structures aged at lower temperature (Figs. 7.15 and 7.22). Thus, we may expect that β particles formed at higher aging temperature are large enough to inhibit dislocations from moving. However, observations of the interactions between β particles and dislocations are indirect in this study, making it uncertain if γ/β interfaces are more resistant to dislocation motion than the γ/α_2 interfaces. *In situ* TEM study of deformation structure during primary creep of FL structures will provide a better understanding of the primary creep behaviour of these microstructures. For such an investigation, a TEM specimen stage capable of *in situ* heating and *in situ* loading would be required.

7.4 Activation energy for creep

As mentioned in section 2.4, creep of two-phase near γ -TiAl alloys has a strong dependence of creep rate on temperature. Such dependence can be described by the empirical creep equation:

$$\dot{\epsilon}_{\min} = A \sigma^n \exp(-Q/RT) \quad \dots\dots (2-1)$$

where $\dot{\epsilon}_{\min}$ is the minimum creep strain rate. The temperature dependence of the creep rate described by this equation reflects the fact that creep involves thermally activated processes and thermally activated mechanisms operating on the atomic scale. If one of these mechanisms dominates and thus controls the creep rate, the activation energy, Q , is identical with the activation energy of this mechanism, or is at least in an immediate relation to it. For instance, in diffusional creep the diffusion of major element(s) in the alloy controls the creep rate and thus the activation energy for creep is very close to that for diffusion of element(s).

The activation energy for creep of FL structures is obtained by determining the steady-state or minimum creep strain rate of FL structures at 207 MPa and temperatures between 720°C and 780°C. Creep tests require hundreds of hours to ensure secondary creep is reached (Table 7.3). The Arrhenius plot of the secondary creep strain rate (Fig. 7.30) gives an activation energy of $Q \approx 391$ kJ/mol for the unaged FL structure, while the activation energy for the FL structure aged at 850°C for 24 hours is about 387 kJ/mol. These values are quite close to each other, and they are comparable to those determined by other researchers [9,93,110,131-134,143-145], but are much higher than those for Ti diffusion in γ -TiAl, 250 – 291 kJ/mol [146-150]. This suggests that diffusion of Ti in FL structures may not be the dominant factor that controls the steady-state creep.

Previous results show that activation energy for creep depends on both microstructure and applied stress, as discussed in section 2.4. Moreover, it is likely that alloying elements also influence the activation energy for creep. Table 7.4 lists activation energies of lamellar structures with additions of different alloying elements. In general,

lamellar γ -TiAl alloy with additions of small amount of elements, such as Cr, Mn, and Nb, will not have a significant change in activation energy for creep at relatively low stress levels [9,93,110,132]. This suggests that the activation energy for creep may not be related to the diffusion of these solute atoms, since Ti and Nb have different diffusion activation energies [149,150,165,166] in Ti-Al system but creep activation energies for the TiAl binary and TiAl-MnNb systems [110,134,145] are nearly the same. However, higher creep stresses and/or excessive amounts of these elements will result in an increase in activation energy for creep [132,143,144]. Additions of elements such as Si, Mo and W lead to an increase in activation energy [9,94,131,133,143,144]. The activation energy for creep of this work agrees with this trend.

Table 7.3 Steady-state/minimum creep rate ($\dot{\epsilon}_{\min}$) of TiAl-WMoSi at various temperatures and 207 MPa

	temperature (°C)	creep time (hr)	$\dot{\epsilon}_{\min}$ (mm/mm/hr)
FL. unaged.	780	630	3.0×10^{-5}
	760	1400	$(0.9 - 1.2) \times 10^{-5}$
	740	1460	4×10^{-6}
	720	1580	2×10^{-6}
FL. aged at 850°C/24hrs.	780	440	2.8×10^{-5}
	760	1200	$(1 - 1.2) \times 10^{-5}$
	740	1680	4×10^{-6}
	720	1630	2×10^{-6}

As mentioned in section 2.4, previous analyses of activation energy for creep were mostly based on the comparison of activation energies between creep and diffusion of Ti in γ -TiAl, and the activation energy for creep is usually greater than for Ti diffusion (Table 7.4). Nevertheless, considering that solute atoms with lower diffusivity may control the diffusion process in solids, it is possible that thermally assisted steady-state

creep is controlled by the diffusion of Al, since the diffusivity of Al is much lower than for Ti at temperatures below 1123°C in Ti-Al alloys [150,165]. Thus, the activation energy for creep of lamellar structure is comparable to that for Al diffusion in the binary γ -TiAl alloy (Table 7.4), as $Q_{Al} = 360$ kJ/mol in γ phase [150] and $Q_{Al} = 395$ kJ/mol in the α_2 phase [165]. It can be postulated that the diffusion of Al along lamellar interfaces controls the steady-state creep, while higher diffusivity of Al can be expected at interfaces than within the grains. Nb has a lower diffusion activation energy (339 kJ/mol [166]), compared to Al, hence will not increase the activation energy for creep [110]. Mn and Cr may play similar roles. Moreover, higher creep activation energies of lamellar near γ -TiAl with additions of Mo, W and Si may be associated to the lower diffusivity of W and Mo [36,53] and the formation of silicide particles [9].

Table 7.4 Activation energies for creep of lamellar structures

	temperature (°C)	stress (MPa)	Q (kJ/mol)
Ti-48Al [134]	700 – 815	207	350
Ti-49Al [145]	750 – 950	166	349 ± 8
		222	373 ± 23
		277	363 ± 47
		333	365 ± 36
Ti-48Al-1.5Cr [132]	700 – 900	200 – 400	292 – 630
Ti-48Al-1.5Cr [9]	690 – 838	300	338
Ti-47Al-2Mn [93]	700 – 900	100	317.2
		130	306.9
		170	364.6
Ti-48Al-2Mn-2Nb [110]	760 – 871	172.3	335
Ti-48Al-1.5Cr-0.26Si [9]	690 – 838	300	388
Ti-48Al-1.5Cr-0.65Si [9]	690 – 838	300	389
Ti-46.6Al-1.4Mn-2Mo [131]	775 – 900	150	390
		200	370
Ti-46.5Al-2Cr-3Nb-0.1W [143]	670 – 720	220 – 380	450
Ti-45.5Al-2Cr-2.6Nb-0.17W-0.1B-0.2C-0.15Si [144]	760 – 920	172	380
Ti-49.1Al-2.2W [133]	750 – 900	172.4	370
Ti-49Al-2W [94]	700 – 900	345	400
Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si, this work	720 – 780	207	≥387

This indicates that during creep, dislocation segments are pinned by lamellar interfaces and further movement of these pinned dislocations depends on the diffusion of the atoms with lower diffusivity at lamellar interfaces. In binary Ti-Al system, diffusion of Al at the pinned dislocation tips controls the steady-state creep rate and therefore, activation energy for creep in binary systems should be equal to or lower than that for solute diffusion of Al. In near γ -TiAl alloys with alloying additions, the alloying elements may influence the diffusion at lamellar interfaces by affecting the diffusivity of Al atoms and/or by controlling the diffusion process themselves. In the former, alloying elements (Cr, Mn and Nb) will affect the Al diffusion rate at lamellar interfaces slightly, resulting in an activation energy for creep close to that for Al diffusion. For the latter, alloying atoms (Mo, W and Si) may dominate the diffusion process by forming more saturated solution or precipitation particles enriched in these alloying elements, leading to an activation energy for creep higher than that for Al diffusion at lamellar interfaces.

The activation energies for creep (≥ 387 kJ/mol) are close to that for β formation (366 kJ/mol) determined in section 6.2, and the formation of β is controlled by the diffusion of W and Mo at lamellar interfaces. This suggests that the higher activation energies for creep between 720°C and 780°C in this alloy ($Q \geq 387$ kJ/mol), compared to that for diffusion of Ti in γ -TiAl (250-291 kJ/mol), are due to the diffusion of W and Mo at lamellar interfaces. It is likely that when dislocations are pinned by γ/α_2 and γ/β interfaces in TiAl-WMoSi alloy (Figs. 7.15, 7.16b and 7.24b), further movement of these dislocations is controlled by the diffusion of W and Mo at these interfaces.

7.5 Summary

The unaged fully lamellar (FL) structure, produced by homogenization + solution heat treatment, yields lower tensile strength and ductility at both ambient and elevated temperatures, compared to the cast duplex structure. Aging the FL structure leads to increased tensile strength as a result of β precipitation during aging. However, the tensile strength and ductility of the aged FL structures are still lower than those of the cast duplex structure. The decreased tensile strength and ductility of FL structures are probably due to the increased lamellar grain size and the cleavage fracture mode of lamellar structure, compared to the cast duplex structure. β particles formed during aging enhance the tensile strength at both ambient and elevated temperatures, but, they do not lead to an increased tensile ductility.

On the other hand, FL structures are more resistant to creep, compared to the cast duplex structure, since the lamellar structure is more resistant to deformation under the creep condition. Deformation of FL structures during creep proceeds primarily by dislocation generation and movement via the deformation of lamellar grains and the deformation of equiaxed γ grains along lamellar grain boundaries. FL structures fractured predominantly by void/cavity formation along lamellar grain boundaries due to the difference in deformability among the adjacent constituents, and by cleavage along lamellar interfaces. Aging the FL structures results in increased resistance to primary creep owing to β precipitation. The decreased creep life of aged FL structure is considered to be a result of rather unstable α_2 plates during creep after aging at elevated temperatures.

The lamellar TiAl-WMoSi has an activation energy for creep of about 390 kJ/mol, which is higher than that for binary TiAl and TiAl-CrNb systems. The higher activation energy of this alloy may be attributed to the lower diffusivity of W and Mo at γ/α_2 and γ/β interfaces. In addition to γ/α_2 interfaces inhibiting dislocation movement, β particles along lamellar interfaces act as obstacles to dislocation motion during the creep thereby increase creep resistance.

Therefore, a heat treatment consisting of homogenization and solution treatment, followed by furnace cooling + air cooling along with subsequent aging at relatively high aging temperature, is suitable for enhanced tensile strength and primary creep resistance.

8 Summary, Conclusions and Suggestions for Future Work

In the past two decades, numerous efforts have been made to understand the microstructure/property relationships, and to improve mechanical properties and creep resistance of near γ -TiAl intermetallic alloys, due to their attractive properties such as low density, high melting temperature, good elevated-temperature strength and modulus retention, high resistance to oxidation and hydrogen absorption, and excellent creep properties. It has been widely acknowledged that a fully lamellar structure with small grain size and finer interface spacing as well as serrated grain boundaries will have a balanced set of properties such as strength, ductility, fracture toughness, and creep resistance. In addition, the fully lamellar structure with fine precipitate particles along the lamellar interfaces was suggested to have enhanced creep resistance and high temperature strength. Moreover, cast gamma based alloys are more competitive for certain applications than those made by wrought or powder metallurgy methods due to the low cost of the casting process. However, the influence of precipitation on mechanical properties of near γ -TiAl intermetallic alloys was not well understood and as-cast titanium aluminides have unacceptably low ductility and strength for many applications, due to the coarse and nonuniform cast lamellar structure that is not readily removed by HIPing.

8.1 Summary

The results of this thesis contribute to existing knowledge of γ -TiAl intermetallic alloys through the examination of an investment cast TiAl-WMoSi alloy. The specific results from this thesis are summarized as follows:

1) Short time solution treatment in the α phase region produces a lamellar structure with some undissolved β particles in the cast bar. Therefore, a heat treatment composed of long time homogenization heat treatment at relatively lower temperature (1300°C/20 hrs) and short time solution treatment in the α phase region (1380°C/20 min) was developed to obtain a uniform fully lamellar (FL) structure. It is difficult to produce a FL structure with small lamellar grain size owing to the large columnar grains in the cast bars.

2) Air cooling after solution treatment results in the formation of Widmanstätten γ structure (γ_w) and massively transformed γ structure (γ_M), while furnace cooling leads to coarse lamellar plates and β precipitation. A two-step furnace cooling + air cooling technique was developed and applied to obtain a γ_w/γ_M -free fully lamellar structure with finer lamellar interface spacing, serrated/interlocked lamellar grain boundaries, and minimum amount of β particles.

3) Aging at intermediate temperatures ranging from 760°C to 1100°C produces both β (ordered BCC structure) and silicide (hexagonal) particles, with the majority of the precipitates being β particles. Silicide precipitation increases as temperature increases. However, the growth of silicides is very slow, compared to β precipitation. β particles formed at temperatures below 850°C exist preferably in the form of thin platelets uniformly distributed in the FL structure, while those formed at higher temperatures appear as ellipsoids unevenly distributed in the lamellar grains

4) The growth rate of β particles increases as aging temperature increases up to 850°C, giving an activation energy for β growth of 366 kJ/mol between 760°C and 850°C. At high aging temperature (950°C), β particles grow very fast at the very

beginning of aging, and then the growth rate of β particles slows down. It is suggested that at low aging temperatures the growth of β particles undergoes a precipitation process controlled by diffusion of W and Mo within the α/γ interface areas. At temperatures above 900°C, β particles grow very fast at first, probably due to the 'short circuit' type diffusion, or the heterogeneous β formation within the areas rich in W and Mo controlled by short-range diffusion. The subsequent β growth proceeds at a much lower rate controlled by the substitutional diffusion of W and Mo from the surrounding areas that were not initially rich in W and Mo.

5) Precipitation hardening at temperatures below 800°C appears to be very slow as aging time increases. Overaging occurs when extended thermal exposure is applied to the lamellar structure especially at higher aging temperature, showing optimum precipitation hardening near 850°C for a reasonably short time.

6) FL structures have lower tensile strength and ductility at both ambient and elevated temperatures, compared to the cast duplex structure. Aging the FL structure leads to increased tensile strength however, the tensile strength and ductility of the aged FL structures are still lower than those of the cast duplex structure. Aging increases the tensile strength of the FL structures at both ambient and elevated temperatures by introducing β and silicide precipitation. However, the appearance of β particles does not lead to an increased tensile ductility.

7) FL structures are more resistant to creep, compared to the cast duplex structure, since the lamellar structure is more resistant to deformation under the creep condition. Deformation of FL structures during creep proceeds via the deformation of lamellar grains by dislocation generation and movement, as well as the deformation of equiaxed γ

grains along lamellar grain boundaries by dislocation movement and twinning. Both lamellar interfaces and β particles act as obstacles to dislocation movement during the creep. FL structures fracture predominantly by void/cavity formation along lamellar grain boundaries and cleavage along lamellar interfaces, due to the difference in deformability between the adjacent constituents.

8) Aging the FL structures results in increased resistance to primary creep due to β precipitation. The decreased creep life of aged FL structures is considered to be a result of rather unstable α_2 plates during creep after aging at elevated temperatures.

9) The lamellar TiAl-WMoSi alloy with β precipitates has an activation energy for creep of about 390 kJ/mol. The relatively higher creep activation energy of this alloy, compared to lamellar binary TiAl and TiAl-CrNb systems, is suggested to be a result of lower diffusivity of W and Mo at γ/α_2 and/or γ/β interfaces. In addition to γ/α_2 interfaces inhibiting dislocation movement, β particles along lamellar interfaces act as obstacles to dislocation motion during creep, thereby increasing creep resistance.

From the these results we may suggest that a heat treatment consisting of homogenization and solution treatment, followed by furnace cooling + air cooling along with subsequent aging at relatively high aging temperature, is suitable for enhanced tensile strength and primary creep resistance.

8.2 Conclusions

This thesis provides an improved basic understanding of the microstructural development, precipitation process, tensile and creep behaviour of an investment cast near γ -TiAl alloy. Specifically the following conclusions are demonstrated:

i) a uniform relatively fine grained fully lamellar microstructure can be developed in investment cast Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si (at%) near γ -TiAl alloy by the application of a heat treatment process consisting of a homogenization heat treatment and solution heat treatment;

ii) precipitation of fine dispersed W- and Mo-rich β particles and titanium silicide particles occurs at lamellar interfaces of the fully lamellar microstructure by introducing a subsequent aging treatment at intermediate temperatures;

iii) the fully lamellar microstructure has decreased tensile strength and ductility at both ambient and elevated temperatures, but an enhanced resistance to creep and an extended creep life, compared the cast duplex microstructure;

iv) precipitation of β and titanium silicide particles in the fully lamellar structure generally has a positive effect on increasing the tensile strength and enhancing resistance to primary creep, however, it reduces the creep life of the fully lamellar structure; and

v) creep deformation of the fully lamellar microstructure proceeds by the deformation of lamellar grains via dislocation generation and movement, as well as the deformation of equiaxed γ grains along lamellar grain boundaries via dislocation movement and twinning.

8.3 Suggestions for future work

This thesis work shows a positive effect of precipitation on both tensile strength and primary creep resistance of the fully lamellar structure. However, the FL structure developed in this study is coarser than those produced by thermal mechanical processing (TMP) and thermal mechanical treatment (TMT) and thus the overall tensile strength is

rather low, compared to the cast duplex microstructure. Investigation of precipitation strengthening the TMP-produced FL structure will be of interest since TMP can also produce a precipitate-free fully lamellar structure with very small grain size. It can be expected that the fine-grained fully lamellar structure produced by TMP with precipitate particles may have a tensile strength comparable to the cast duplex microstructure and a superior primary creep resistance, which may be beneficial for gas turbine blade applications.

The instantaneous creep strain is related to the tensile behaviour of the microstructure at the same temperature: the microstructure that is more resistant to tensile deformation, i.e. higher Young's modulus and/or higher yield strength, will have a lower instantaneous creep strain. Creep tests of the fully lamellar structures at a relatively lower stress (<130 MPa) and determination of Young's modulus may be helpful to further understand the relationship between primary creep resistance and tensile behaviour of this microstructure. Moreover, *in situ* TEM investigation of the deformation structures during primary creep will explain why the FL structure aged at higher temperature is more resistant to primary creep.

The creep rate is probably controlled by the diffusion of alloying elements at the lamellar interfaces in the FL structure with fine lamellar interface spacing. Determination of activation energy for creep of Ti-Al-W system may be of interest since a creep activation energy similar to TiAl-WMoSi alloy can be expected, if the diffusion of alloying elements does play a role in controlling creep rate. Also, investigation of creep activation energy of a fully lamellar TiAl-WMoSi alloy with much wider lamellar interface spacing might provide insight in understanding the relation between creep rate

and diffusion at lamellar interfaces. In FL structure with only wide lamellar plates, dislocation movement becomes easier, compared to the FL structure with fine lamellar interface spacing, due to the decreased obstacles to dislocation motion. Therefore, a lower creep activation energy may be expected since dislocation glide may dominate the creep process while the diffusion of alloying elements controlling steady-state creep becomes less significant.

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Figures

Fig. 2.1 Binary Ti-Al phase diagram [27].

Fig. 2.2 γ -TiAl has an ordered face centred tetragonal (L10) structure (a) and α_2 -Ti₃Al has a complex hexagonal (DO₁₉) structure [27].

Fig. 2.3 Estimated orientation relationship between ω (hexagonal) and β (B2) phases [30].

Fig. 2.4 Typical microstructures of near γ -TiAl intermetallics [44]: (a) fully lamellar, (b) near lamellar, (c) duplex and (d) equiaxed γ structure.

Fig. 2.5 Effect of cooling rate on microstructure after solution treatment in α phase field (a) 210°C/s, lamellar, (b) 240°C/s, lamellar + γ_w + γ_M , (c) 430°C/s, γ_M and (d) 650°C/s, γ_M + α_2 [46]. Ti-48Al.

Fig. 2.6 Proposed CCT diagram for Ti-48Al alloy [46]. γ_{Ls} and γ_{Lf} are temperatures at which lamellar structure starts and finishes, respectively. γ_{ws} and γ_{Ms} are temperatures at which γ_w and γ_M start to form.

Fig. 2.7 Morphologies of β (B2) phase in near γ -TiAl intermetallics [14.38]:
 (a) β (B2) blocks. Ti-44Al-2Mo.
 (b) β rods, and
 (c) β precipitates. Ti-47Al-2W-0.5Si.

Fig. 2.8 Titanium silicides in near γ -TiAl intermetallics formed during casting (a) and annealing (b) [16].

Fig. 2.9 Effect of lamellar structure on tensile properties [56]. Ti-48Al-3Cr and Ti-48Al-2Cr-2Nb.

Fig. 2.10 Effect of lamellar structure on fracture toughness of near γ -TiAl [25].

Fig. 2.11 Effect of lamellar orientation on tensile properties [59]. ϕ is the angle between lamellar interface and stress direction. Ti-49.3Al.

Fig. 2.12 Effect of lamellar structure on creep behaviours of near γ -TiAl [56,64].

Fig. 2.13 Effect of lamellar interface spacing on creep behaviours of TiAl [51].

Fig. 2.14 Small amount of silicon enhances creep resistance of near γ -TiAl [94].

Fig. 2.15 Deformation transfer across an α_2/γ interface [121].

Fig. 2.16 Deformation transferred across the lamellar interfaces as strain increases [57]: (a) $\epsilon = 0.3\%$ and (b) $\epsilon = 1.21\%$.

Fig. 2.17 Nucleation of a microcrack at grain boundary in fully lamellar structure [119].

Fig. 2.18 Deformation structures in (a) "soft-oriented" and (b) "hard-oriented" lamellar grains [125].

Fig. 2.19 Pinning of dislocation segments by precipitation particles [127].

Fig. 2.20 Deformation across lamellar interfaces accompanied by void formation (a) and recrystallization of γ grains at lamellar grain boundary (b) and γ grain boundary (c) at elevated temperatures [112,113,125].

Fig. 2.21 Generation of dislocation loops (indicated by arrow 2) at a semicoherent α/γ interface during *in situ* heating [130]: 300K $\rightarrow$ 994K/30min $\rightarrow$ 300K.

Fig. 2.22 Dislocations slip across γ/γ interfaces providing another source for deformation [87].

Fig. 3.1 Geometry of tensile and creep samples, dimensions in inches.

Fig. 3.2 Repeating creep tests of samples of same condition show a good reproducibility of creep curves up to 800 hours with a creep strain difference less than 10%.

Fig. 4.1 Cross section of the cast bar of Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si.

Fig. 4.2 Cast and HIPped columnar lamellar structure in the bottom portion of the cast bar (a) and fine equiaxed grains in the top centre portion (b).

Fig. 4.3 As-cast and HIPped β phase in lamellar grains.

Fig. 4.4 Fewer β particles, white areas in SEM backscattered image, appear in the top section (a) of the cast bars, compared to in the bottom section (b).

Fig. 4.5 Image analysis of SEM backscatter images shows a lower density of heavier alloying elements, as evident by lower fraction of cast β phase within the top portion of the cast bars.

Fig. 4.6 No significant compositional difference of Ti and Al along the cast bar can be observed from SEM-EDS semi-quantitative analysis.

Fig. 5.1 Solution treatment at 1380°C for 20 minutes produces a nearly lamellar structure with some undissolved β particles.

Fig. 5.2 A heat treatment consisting of a homogenization treatment below T_α and a solution heat treatment above T_α followed by an aging treatment below T_h should be applied to the investment cast near γ -TiAl alloys, in order to obtain a fully lamellar structure.

Fig. 5.3 Cast duplex structure after homogenization treatment at 1010°C for 50 hours.

Fig. 5.4 Microstructures after homogenization heat treatments in the $\alpha_2 + \gamma$ phase region (a) 1280°C/20 hours, (b) 1300°C/20 hours and (c) 1320°C/2 hours. Significant grain growth starts at 1320°C.

Fig. 5.5 TiAl-WMoSi, homogenized at 1300°C for 20 hours followed by (a) 1360°C/30 minutes, bottom portion, (b) 1370°C/30 minutes and (c) 1380°C/20 minutes, top portions. Samples were furnace cooled to 1220°C followed by air cooling. Undissolved γ grains in (b) suggest that α transus temperature varies along the cast bar.

Fig. 5.6 Relatively uniform grain size can be obtained after $1180^{\circ}\text{C}/100\text{hrs} + 1300^{\circ}\text{C}/20\text{hrs} + 1360^{\circ}\text{C}/30\text{min}$ within the bottom portion of the cast car (a), whereas heat treatment at $1300^{\circ}\text{C}/20\text{hrs} + 1380^{\circ}\text{C}/20\text{min}$ leads to nonuniform lamellar grains in the top portion (b).

Fig. 5.7 No evidence shows β existing after 1300°C/20 hours + 1380°C/20 minutes. XRD results.

Fig. 5.8 Air cooling after solution treatment produces a mixed structure of lamellar grains, Widmanstätten γ and massively transformed γ structure, γ_M , (a), with some β precipitation along the lamellar/Widmanstätten γ (b) and γ_M/γ_M boundaries and inside γ_M (c).

Fig. 5.9 After furnace cooling from α phase region, (a) β particles can be occasionally observed along lamellar interfaces in the top portion of the cast bar, TEM BF image, and (b) β particles inside lamellar grains and along grain boundaries in the bottom portion, SEM backscatter image.

Fig. 5.10 α_2 has higher solubility of W and Mo. W and Mo are unevenly distributed in α_2 plates inside the lamellar structure. TEM-EDS, convergent beam, spot size of 2 nm.

Fig. 5.11 SEM backscatter image shows that α_2 plates are brighter than γ plates, resulting from the higher solubility of W and Mo.

Fig. 5.12 β particle precipitation can hardly be observed along α_2/γ_M boundaries or inside γ_M after water quenching (a), while β particles precipitate predominantly along the interface between lamellar and Widmanstätten γ structures during air cooling (b). SEM backscatter images.

Fig. 5.13 After solution treatment, furnace cooling to 1220°C followed by air cooling produces a fully lamellar structure with few dislocations (a) and virtually no β particles (b) in the top portion of the cast bar.

Fig. 5.14 After homogenization and solution heat treatments, furnace cooling to 1250°C followed by air cooling results in the formation of γ_M (a), while no γ_M structure can be found when furnace cooling to 1220°C followed by water quenching (b).

Fig. 5.15 More β particles form in the bottom portion of the cast bars after furnace cooling to 1220°C followed by air cooling in grain boundary area (a) and inside lamellar grain (b). SEM backscatter image.

Fig. 5.16 SEM backscatter imaging shows "clouds" of heavy elements in the lamellar structure. Contrast difference indicates the non-uniform distribution of W and Mo.

Fig. 6.1 β precipitates are enriched in W and Mo, while silicides are almost free of W and Mo. Aged at 1050°C for 24 hours. TEM-EDS, convergent beam, spot size of 2 nm.

Fig. 6.2 Scanning transmission electron microscopic (STEM) images from TiAl-WMoSi:
 (a) bright field image,
 (b) digital x-ray image for W ($L\alpha$),
 (c) digital x-ray image for Mo, and
 (d) digital x-ray image for Si ($K\alpha$) and W (M).
 Aged at 1050°C for 24 hours. The mapped area is 2.86 μm x 2.86 μm .

Fig. 6.3 Precipitation occurs at 760°C and the size and density of particles increase as time increases. a) 24 hours, b) 100 hours, c) 250 hours and d) 400 hours. TEM bright field images.

Fig. 6.4 Finely dispersed precipitates obtained after aging at 800°C for 24hrs (a) and 72hrs (b), and at 850°C for 24hrs (c and d), with most precipitation occurring along the lamellar interfaces. (d) is the same area as in (c), but with sample tilted so that lamellar interfaces are parallel to incident electron beam. Arrows in (c) indicate fine silicide particles at lamellar interfaces. TEM bright field images.

Fig. 6.5 β particles formed after aging at 850°C for 24 hours. (b) is the same area as in (a) but with sample tilted. The width of the β particle indicated by arrow in (b) is greater than the thickness of the α_2 plate that contains this particle, as indicated by arrow in (a), indicating that these β particles are of thin-plate shape.

Fig. 6.6 Precipitates formed after 24 hour aging at 900°C (a), 950°C (b), and 1050°C (c and d). The growth of silicides is very slow, whereas β precipitates appear predominantly in the form of ellipsoidal particles.

Fig. 6.7 Diffraction patterns of silicide particles. Convergent beam, spot size 2 nm.

Fig. 6.8 Diffraction patterns of β precipitates. Electron beam parallel to $[\bar{3}11]_{\beta}$ in (a) and $[\bar{1}10]_{\beta}$ in (b). Convergent beam, spot size 2 nm.

Fig. 6.9 Speculated growth of a thin plate-like β particle at a α/γ interface at temperatures below 850°C .

Fig. 6.10 A plate-like precipitate with dislocations on its broad faces (a) and the schematic (b) of the thickening of such a plate via repeatable step mechanism resulting from the displacement of part of an immobilized interface [163,164].

Fig. 6.11 Maximum projected area of β particles as a function of time.

Fig. 6.12 Arrhenius plots of the growth rates (A/t) of β particles at temperatures between 760°C and 950°C.

Fig. 6.13 Maximum microhardness occurs at 850°C when aged for 24 hours. Before aging, all samples were homogenized and solution treated, followed by FC + AC.

Fig. 6.14 Microhardness increases slowly as the time increasing at 800°C. Before aging, all samples were homogenized and solution treated, followed by FC + AC.

Fig. 6.15 Overaging occurs at above 850°C when extensive thermal exposure was applied to the lamellar structure. Samples were homogenized and solution treated followed by FC + AC, before aging.

Fig. 7.1 Tensile behaviour of cast duplex and FL structures at ambient temperature.

Fig. 7.2 Tensile behaviour of cast duplex and FL structures at 760°C.

Fig. 7.3 The cast duplex structure failed predominantly by translamellar fracture (a) at ambient temperature, with small cleavage facet (indicated by arrow) (b) occurring within lamellar grains.

Fig. 7.4 Fracture surfaces of FL structures after tensile test at ambient temperature: unaged (a) and (b), 850°C/24hr aged (c) and (d), and 950°C/24hr aged (e) and (f). Cleavage is the predominant fracture mode in the FL structures, regardless of the aging treatment. Arrows indicate delamination of lamellae.

Fig. 7.5 At 760°C cast duplex structure exhibits a fracture mode similar to that at room temperature.

Fig. 7.6 FL structures failed predominantly by cleavage during tensile test at 760°C: unaged (a) and (b), 850°C/24hr aged (c) and (d), and 950°C/24hr aged (e) and (f).

Fig. 7.7 Creep curves for cast duplex and FL structures of TiAl-WMoSi. Dotted line is the estimated portion of creep curve for the 850°C/24hr aged FL structure.

Fig. 7.8 Aging the FL structure results in an increased resistance to primary creep.

Fig. 7.9 Cross section of an unaged FL sample fractured after creep at 760°C and 207 MPa.

Fig. 7.10 Void/cavity coalescence along lamellar grain boundaries in area close to fracture surface (a) in unaged FL while the formation of voids/cavities is less where far from the fracture surface (b).

Fig. 7.11 The unaged FL structure during secondary creep, 1.8% strain (a) and tertiary creep (b) shows void/cavity formation during secondary creep and lamellar delamination during tertiary creep.

Fig. 7.12 Lamellar delamination becomes substantial during creep in the FL structures aged at 850°C for 24 hours (a) and at 950°C for 24 hours (b).

Fig. 7.13 The unaged FL structure crept to secondary creep, 1.8% strain (a) and tertiary creep (b). Shear across the lamellar interfaces occurs during tertiary creep as evident by the waved lamellar plates in (b). Arrows indicate share across lamellae.

Fig. 7.14 Shear crossing lamellar interfaces appears during secondary creep (1.8% strain) in the 850°C/24hr aged FL structure but mostly within the lamellar grain boundary areas (a). Further creep deformation leads to shear deformation across lamellar plates (b) during tertiary creep. Arrows indicate share across lamellae.

Fig. 7.15 Dislocations in the unaged FL structure after crept for 24 hours: (a) "hard" and (b) "soft" oriented lamellar grains. Arrow in (b) indicates a dislocation bowed out of γ/α_2 interface.

Fig. 7.16 The number of dislocations increases slowly during secondary creep (1.8% strain). β particles interact with dislocations in (b). "soft" oriented grains. Unaged FL structure.

Fig. 7.17 Higher dislocation density in wider γ plates (a), equiaxed γ grains and lamellae close to these equiaxed γ grains (b). "soft" oriented grains. Unaged FL, 1.8% strain. Dislocation continues to be generated at γ/α_2 interface during secondary creep as indicated by arrow in (a).

Fig. 7.18 During tertiary creep, dislocation density increases within the lamellar grains in "soft" orientation (a), whereas fewer dislocations appear in "hard" oriented lamellar grains (b). Unaged FL structure. Arrows in (a) indicate new dislocations at γ/α_2 interface during tertiary creep.

Fig. 7.19 Interactions between dislocations and β particles (indicated by arrows) in lamellar grain with "soft" orientation. (b) is the same area of (a) but with foil tilted. Unaged FL, tertiary creep.

Fig. 7.20 Interactions between dislocations and β particles (a) and deformation twinning (b) in equiaxed γ grain along lamellar grain boundary. Unaged FL, tertiary creep.

Fig. 7.21 Dislocation is the only type of deformation structure that can be observed in the unaged FL structure until fracture occurs. (a) "hard" and (b) "soft" oriented grains.

Fig. 7.22 Dislocations in the 850°C/24hr aged FL structure after crept for 24 hours: (a) "hard" and (b) "soft" oriented lamellar grains. The arrow indicates a dislocation loop coming out at γ/α_2 interface.

Fig. 7.23 The number of dislocation is quite small during secondary creep (1.8% strain) within "hard" oriented lamellar grain (a), whereas more dislocations appear in the lamellar grain with "soft" orientation (b). 850°C/24hr aged FL structure. The arrow indicates dislocation loops coming out at γ/α_2 interface.

Fig. 7.24 Interactions between dislocations and β particles in lamellar grain with "soft" orientation. (b) is the same area of (a) but with foil tilted. 850°C/24hr aged FL, secondary creep (1.8% strain).

Fig. 7.25 Dislocations in "hard" (a) and "soft" (b) oriented lamellar grains, as well as in equiaxed γ grains at lamellar grain boundaries (c) at the onset of tertiary creep of the 850°C/24hr aged FL structure.

Fig. 7.26 Dislocation activity is the only deformation mode in both “hard” (a) and “soft” (b) oriented lamellar grains in the 850°C/24hr aged FL structure until fracture occurs. Arrows in (b) show dislocation generation at γ/α_2 interface.

Fig. 7.27 Dislocations in the 950°C/24hr aged FL structure after crept for 24 hours: (a) "hard" and (b) "soft" oriented lamellar grains. Arrows in (b) indicate interactions between dislocations and β particles.

Fig. 7.28 Interactions between dislocations and β particles in lamellar grain with "soft" orientation. (b) is the same area of (a) but with foil tilted. 950°C/24hr aged FL, creep fractured.

Fig. 7.29 In "hard" oriented lamellar grains, fewer dislocations appear in γ lamellae far from grain boundaries (a), whereas more dislocations show up in areas close to grain boundaries (b). Deformation twinning can only be found in equiaxed γ grains (indicated by arrow) along grain boundaries. 950°C/24hr aged FL, creep fractured.

Fig. 7.30 The Arrhenius plots of the secondary (steady-state) creep strain rate of FL structures. Activation energies for creep of unaged FL structure and aged FL structure are almost identical.

Appendix 1 Summary of homogenization and solution treatments

Homogenization treatments

temperature (°C)	time (hours)	microstructure	figure
1010	50	cast duplex with fine equiaxed γ grains at the center of the cast bar.	5.3
1140	10,20,40, 168	cast duplex with fine equiaxed γ grains at the center of the cast bar, number and size of γ grains increase with increased time.	
1160	10,20, 40	cast duplex with fine equiaxed γ grains at the center of the cast bar, number and size of γ grains increase with increased time.	
	168	equiaxed γ grains + some lamellar grains.	
1180	5,10,20, 40	cast duplex with fine equiaxed γ grains at the center of the cast bar, number and size of γ grains increase with increased time.	
	70	equiaxed γ grains + lamellar grains.	
	100	equiaxed γ + some lamellar grains.	
1200	5,10,20, 40	cast duplex with fine equiaxed γ grains at the center of the cast bar, number and size of γ grains increase with increased time.	
1220	5,10,20, 40	cast duplex with fine equiaxed γ grains at the center of the cast bar, number and size of γ grains increase with increased time.	
1240	5,10,20	cast duplex with fine equiaxed γ grains at the center of the cast bar, number and size of γ grains increase with increased time.	
1260	5,10,20	cast duplex with fine equiaxed γ grains at the center of the cast bar.	
1280	5,10,20	cast duplex with fine equiaxed γ grains at the center of the cast bar.	5.4a
1300	5,10,20	cast duplex, equiaxed γ grains at the center of the cast bar starts to grow, γ grain size < 80 μm .	5.4b
1320	2	cast duplex, γ grain size of about 200 μm at the center of the cast bar.	5.4c
1340	2	cast duplex, γ grain size of about 400 μm at the center of the cast bar.	
1360	0.5	fully lamellar with grain size of 0.4 – 1.4 mm and some undissolved β particles and γ grains.	
	2	fully lamellar with grain size of 0.8 – 2.5 mm.	
1380	1/3	fully lamellar with grain size of 0.4 – 1.8 mm and some undissolved β particles.	5.1
	2	fully lamellar with grain size of 1 – 3 mm.	

Homogenization + solution treatments followed by furnace cooling

heat treatment	microstructure	figure
1300°C/20hrs + 1360°C/15min	fully lamellar with grain size of 0.4 – 1.2 mm and some undissolved γ grains.	
1300°C/20hrs + 1360°C/30min	fully lamellar with grain size of 0.4 – 1.4 mm within lower portion of the cast bar only.	5.5a
1300°C/20hrs + 1370°C/30min	fully lamellar with grain size of 0.4 – 1.6 mm and some undissolved γ grains within upper portion of cast bar.	5.5b
1300°C/20hrs + 1380°C/20min	fully lamellar with grain size of 0.4 – 1.8 mm.	5.5c, 5.6b
1180°C/100hrs + 1300°C/20hrs + 1360°C/30min	fully lamellar with grain size of 0.4 – 1 mm within lower portion of the cast bar only, grain size is rather uniform across the cast bar.	5.6a

Appendix 2 Summary of aging treatment at intermediate temperatures

temperature (°C)	time (hours)	microstructure	maximum projected area of β particles (μm^2)	figure
760	24	some fine β platelets, few silicide particles.	0.002	6.3a
	100	fine dispersed β platelets, few silicide particles.	0.008	6.3b
	250	fine dispersed β platelets, few silicide particles.	0.01	6.3c
	400	fine dispersed β platelets, few silicide particles.	0.014	6.3d
800	24	some fine β platelets, few silicide particles.	0.003	6.4a
	72	fine dispersed β platelets, few silicide particles.	0.014	6.4b
	216	fine dispersed β platelets, few silicide particles.	0.026	
850	24	fine dispersed β platelets, few silicide particles.	0.012	6.4c 6.4d 6.5
	48	fine dispersed β platelets, few silicide particles.	0.036	
	72	fine dispersed β platelets, few silicide particles.	0.053	
900	24	β platelets and ellipse blocks, few silicide particles.	0.028	6.6a
950	4	β platelets and ellipse blocks, few silicide particles.	0.058	
	12	β platelets and ellipse blocks, few silicide particles.	0.063	
	24	β ellipse blocks and platelets, few silicide particles.	0.079	6.6b
	48	β ellipse blocks, some silicide particles.	0.098	
	96	β ellipse blocks, some silicide particles.	0.107	
	240	β ellipse blocks, some silicide particles.	0.28	
1000	24	β ellipse blocks, some silicide particles.	0.232	
1050	24	β ellipse blocks, some silicide particles.	0.395	6.6c 6.6d

Appendix 3 Summary of creep tests

test condition	sample condition	test time (hour)	note
760°C & 207 MPa	1010°C/50hrs	405	fractured.
	FL unaged	24	interrupted at primary creep.
		650	interrupted at 1.8% strain.
		1745	interrupted at tertiary creep.
		2759	fractured.
	FL 850°C/24hrs	24	interrupted at primary creep.
		1102	interrupted at 1.8% strain.
		1722	interrupted at tertiary creep.
		1798	fractured.
	FL 950°C/24hrs	24	interrupted at primary creep.
		1215	fractured.
		2166	fractured.
780°C & 207 MPa	FL unaged	620	interrupted at secondary creep.
	FL 850/24hrs	440	interrupted at secondary creep.
740°C & 207 MPa	FL unaged	1460	interrupted at secondary creep.
	FL 850/24hrs	1680	interrupted at secondary creep.
720°C & 207 MPa	FL unaged	1580	interrupted at secondary creep.
	FL 850/24hrs	1620	interrupted at secondary creep.