



THESIS

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thesis entitled

High Temperature Creep Deformation of Gamma-Based Titanium Aluminide

presented by

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has been accepted towards fulfillment of the requirements for

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HIGH TEMPERATURE CREEP DEFORMATION OF GAMMA-BASED TITANIUM ALUMINIDE

By

Randy S. Beals

A THESIS

Submitted to Michigan State University in partial fulfillment of the requirements for the degree of

MASTERS OF SCIENCE

Department of Materials Science and Mechanics

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ABSTRACT

HIGH TEMPERATURE CREEP DEFORMATION OF GAMMA-BASED TITANIUM ALUMINIDE

By

Randy S. Beals

As part of an ongoing research concerning creep deformation mechanism(s) responsible for the creep behavior of ingot investment cast near γ -TiAl, a study has been completed which several creep studies where performed and some theoretical arguments concerning the behavior of TiAl where introduced. The theoretical arguments include the combining the composite rule-of-mixture and the power-law Mukerjee-Bird-Dorn equation as a mathematical creep deformation model. Also, a transitional interface between the gamma and alpha2 constituents is introduced. A literature review has been completed and the creep characteristics of different compositions was explored at different test conditions in order to list the trends that appear in the data. The creep experiments achieved where on the alloy Ti-48Al-2Cr-2Nb and the data obtained was compared to the data in the literature. All the experimental data in the literature and from this study was normalized by the temperature dependence of the diffusively and the shear modulus. The temperature normalization of values allows the investigator a more accurate view of the stress dependence on the creep deformation. The creep characteristics where then analyzed to improve our understanding of the deformation mechanism(s) responsible for the creep behavior of TiAl.

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CHAPTER ONE

INTRODUCTION

Titanium aluminide TiAl based materials are promising candidates for high temperature service. This is due to their high specific strength, stiffness at elevated temperatures, and good oxidation and creep resistance. Also, their density is 2.5 times lower than current nickel-based superalloys. However, very little research of near γ -TiAl based alloy creep behavior has been accomplished, and the creep deformation mechanisms are not clearly identified. The activation energies reported are much larger than those for self-diffusion and interdiffusion in TiAl, which implies that the creep rate may be controlled by processes other than the usual lattice diffusion mechanism. Also, the value of the stress exponent varies widely from about 2 to 8 in the literature. This suggests that several deformation mechanisms are not known and the limited results in the literature are not always consistent with the common creep theories.

This work is concerned with studies of the high temperature creep behavior of gamma-based TiAl alloys having the duplex microstructure. A main emphasis was addressed toward the composition of Ti-48Al-2Cr-2Nb (at%). The creep characteristics of different compositions and microstructures was compared in order to find possible trends that appear in the data. The creep properties that where investigated were the activation energy for diffusion, Q, and the stress exponent, n, in order to determine what role they play in the creep behavior of the material.

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CHAPTER TWO

THEORY

2.1 REVIEW OF CREEP

2.1.1. The high-temperature materials problem

It is well known to material scientists that the strength of most metals decreases with increasing temperature. Since the mobility of atoms increases rapidly with temperature, it can be appreciated that diffusion-controlled processes can have a very significant effect on high-temperature mechanical properties. High temperature will also result in greater mobility of dislocations by the mechanism of climb. The equilibrium concentration of vacancies likewise increases with temperature. New deformation mechanisms may come into play at elevated temperatures. In some metals the slip system changes, or additional slip systems are introduced with increasing temperature. Deformation at grain boundaries becomes an added possibility and grain boundary motion or recrystallization may occur during the high-temperature deformation of metals. Another important factor to consider is the effect of prolonged exposure at elevated temperature on the metallurgical stability of metals and alloys.

Thus, it should be apparent that the successful use of metals at high temperatures involves a number of problems. Aggressive alloy-development programs have produced a number of materials with improved high-temperature properties, but the

ever increasing demands of modern technology require materials with even better hightemperature strength and oxidation resistance. Since the introduction of the gas-turbine engine, materials must operate in critically stressed parts, such as turbine buckets, at temperatures around 765°C (1400 °F).

An important characteristic of high-temperature strength is that it must always be considered with respect to some time scale. The tensile properties of most engineering metals at room temperature are independent of time, for practical purposes. It makes little difference in the result if the loading rate of a tension test is such that it requires 2 hours or 2 minutes to complete the test. However, at elevated temperatures the strength becomes very dependent on both strain rate and temperature. A number of metals under these conditions behave in many respects like viscoelastic materials. A metal subjected to a constant tensile load at an elevated temperature will creep and undergo a timedependent increase in length. A strong time dependence of strength becomes important for different materials at different temperatures. What is high temperature for one material may not be so high for another. To compensate for this a high temperature is generally regarded as greater than one-half the melting temperature $(0.5 T_m)$.

2.1.2. Stages of creep

Creep is defined as "the resistance of materials to deformation and failure over long times, under load and at high temperatures"[1]. An example of a simple creep situation can be expressed as an copper wire that is loaded at one end by a large weight, and at the other end is attached to the ceiling. The wire is then heated and a time-dependent deformation occurs.

The time-dependent creep deformation is usually thought of as being divided into three stages: Primary, secondary and tertiary creep(See Figure 2.1).

Stage one (*primary*, or *transient*) creep is actually divided into two stages. The first consists of an value of ε_0 which is the virtually instantaneous increase in length due to the elastic and plastic deformation which occurs on loading. The second consists of an value of ε_c which is the time-dependent increase in length due to creep deformation. Materials subjected to transient creep shows that the structure changes with increasing strain in a manner somewhat analogous to that observed during work hardening; e.g., the dislocation density increases and, in many materials, a dislocation subgrain structure is formed with a cell size that decreases to a steady-state value with increasing strain. These structural changes are consistent with the creep rate decreasing with increasing time in stage I creep. In Great Britain, they divide stage one into the ε_0 and ε_c components and combine them with the other two stages, and thus having four stages of creep.

Stage two (*secondary*, or *steady-state*) creep is characterized by a constantcreep-rate. The amount of deformation strain divided by the time, (dɛ/dt) known as the *strain rate* or the *creep rate*, is a constant for this creep region. The invariant microstructure is indicative that recovery effects are concurrent with deformation during Stage II; that is, in the absence of recovery the dislocation density would increase and a subgrain structure, if present, would become progessively finer with increasing strain. Hence, harding mechanisms effective at low temperatures are not so useful at higher temperatures, and stage II creep can be viewed as a regime in which the intrinsic work-hardening capacity of the material is balanced by recovery or "softening" effects allowed by the increased deformation temperature. Because of this relation between the material deformation and the

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strain-rate being a constant, amost all of the mechanical modeling of creep deformation mechanisms is determined in the steady-state creep regime.

Subsequent to stage II creep, stage III (*tertiary*) creep is observed. During tertiary creep, the creep rate is greater than stage II and increases continuously. This is shown further in a plot of strain vs. time (Figure 2.1), which also shows clearly that stage II is characterized by a constant, minimum creep rate. The accelerating creep deformation of stage III eventually leads to material fracture and is related to several factors. Some of these factors include the onset of recrystallization, the coarsening of second-phase particles and/or the formation of internal cracks or voids.



Figure 2.1 Characteristic three stage creep curve.

2.1.3. Temperature effects

Earlier, creep was described as being a time-dependent deformation mech-

anism; but it is also temperature-dependent. The "constant" steady-state strain rate

increases exponentially with increasing temperature (Figure 2.2).



Figure 2.2 Increasing strain rate with increasing temperature.

By measuring the secondary creep rates recorded at the same stress, but at different temperatures, one can investigate the relationship

 $\ln (d\epsilon/dt)_{ss} \propto \exp(-Q/RT)$

where (dɛ/dt)_{ss} is the steady-state strain rate, Q is the activation energy for creep (diffu-

sion), R is the universal gas constant (8.31 J/mol*K), and T is the temperature. By plotting ln (dɛ/dt) vs. (1/T) one can often obtain a good linear relationship, that yields the Q value







When procedures of this type have been used to study the creep behavior of pure metals at high temperatures, the values of Q have been found to be close to the activation energy for lattice self-diffusion. This emphasizes the importance of diffusion under the high-temperature creep conditions.

2.1.4. Stress effects

Another aspect of creep is the dependence on the applied stress. So, the "constant" steady-state rate increases exponentially with increasing stress very similarly to it's response to an increase in temperature(See Figure 2.4).



time

Figure 2.4 Increasing strain rate with increasing stress.

Similar to the last relationship discussed, by measuring the secondary creep rates recorded at the same temperature, but at different stresses, one can investigate the relationship:

$$\log (d\epsilon/dt)_{ss} \alpha n \log \sigma$$

where (d ϵ /dt) is the steady-state strain rate, n is the stress sensitivity exponent and σ is the

applied stress(See Figure 2.5)





Figure 2.5 Plot of stress exponential.

2.1.5. Power law relation

This brings us to the discussion of the "power law" relationship which is widely used to describe high temperature creep. The power law representation of high temperature creep is defined by the equation below[2]:

$$(d\varepsilon/dt)_{ss} = A\sigma^{n} exp(-Q/RT)$$
(1)

which is usually plotted in the form of log $(d\epsilon/dt)_{ss} = \log A + n\log\sigma - Q/RT$

where $(d\epsilon/dt)_{ss}$ is the steady-state strain rate, A is an material constant, σ is the applied stress, n is the stress sensitivity exponent, Q is the activation energy, R is the universal gas constant, T is the temperature.

2.1.6. Transition in the stress exponent

However, the power law representation is accurate in limited temperature and stress regimes. That is, while n is approximately a linear constant over much of the stress range, the plot curves upward at the higher stresses. So at very high stresses, the curvature of the line suggests that n increases continuously with increasing stress. This increase in the n value with increasing stress is referred to as *power law breakdown*.

The steady-state is the most important condition for creep modeling. However, our understanding of the creep behavior is obscured by the n value changing during creep. This has been discovered when the plot of $log(d\epsilon/dt)_{ss}$ vs. $log\sigma$ have been found to curve with decreasing applied stress, such that a transition occurs from n = 4 at high stresses to n = 1 in the low stress regime(See Figure 2.6).



Figure 2.6 Transition of n with different stress conditions.

2.1.7. Transition in activation energy

A similar transition occurs to the activation energy, Q, but instead of different stress regimes, this transitions occurs at different temperature regimes. Creep at high temperatures (0.7 T_m) frequently has the activation energy equal to that of self-diffusion. Creep properties studied at lower temperatures (0.4 T_m) have found that the activation energy equals about 1/2 that of self-diffusion. This transition from the activation energy

value equal to the self-diffusion to a value of 1/2 the self-diffusion activation energy has been found regardless if the test carried out at high stresses (n = 4 - 6) or low stresses (n = 1).

The observation of the activation energy that suddenly changes from one value to another results from "preferential diffusion paths" to account for the change. [3]

To explain further, the magnitude of the activation energy for diffusion depends on the bond energies between the moving atom and it's neighbors. Because the surface (or interface, grain boundary) atoms have fewer neighbors and therefore, fewer bonds than the atoms in the crystal, surface paths offer an "easier path" for diffusion. Grain boundaries and the core of the lattice dislocations define regions where the atomic arrangements are less regular than in the crystal lattice, and should provide an "easier path" characterized by activation energies less than the value of lattice diffusion(See Figure 2.7).



(1/RT)

Figure 2.7 Transition of Q with different temperature conditions.

At high temperatures when lattice diffusion is rapid, diffusion along the easy paths makes a negligible contribution to the overall diffusion rate, because the volume of the grain boundaries is small when compared to the total volume of the crystalline sample. On the other hand, as bulk diffusion rates decrease, the diffusion along the easy paths becomes progressively more important since the rate of the process with the lower activation energy changes less with decreasing temperature. Therefore, the process associated with the high activation energy value is dominant at high temperatures, but the process having the lower activation energy becomes dominant at lower temperatures.

2.2. CREEP DEFORMATION MECHANSIMS

As mentioned, creep can be viewed phenomenologically as a process in which work hardening and recovery processes occur on the same time scale as the deformation process. In this section, several mechanisms of creep are discussed to illustrate the basis of the power law creep equation (Eqn. 1). Depending on temperature and applied stress, dislocation glide, dislocation recovery (for example, by processes involving dislocation climb), or other diffusional-flow mechanisms may dominate creep deformation. Several of the mechanisms described, particularly those related to dislocation climb, are speculative in that they can not be (or at least have not yet been) verified by direct microstructural examination. Nonetheless, processes similar to the ones investigated surely occur during creep deformation. Beyond that, a description of the processes allows the correlation of the power-law equation (Eqn. 1) with microstructure and the applied external "forces" of temperature and stress. <u>.</u>

Generally, deformation mechanisms that occur during creep are usually divided into two main categories: diffusion controlled mechanisms and dislocation controlled mechanisms.

2.2.1. Diffusion controlled creep mechanisms

There are two main types of diffusion controlled creep mechanisms: Nabarro-Herring and Coble creep processes.

(a) Nabarro-Herring Creep

Nabarro-Herring creep is a process that involves diffusional transport of atoms through the grain. Consider the example of a grain of a polycrystalline deforming at high temperatures under stress(See Figure 2.8).

If vancancies are generated at the boundaries experiencing the tensile stress and these vacancies diffuse to the boundaries under compression. This results in a flow of vacancies and a counter flow of atoms.



Figure 2.8 Schematic of Nabarro-Herring creep on an ideal grain, after [4].

For Nabarro-Herring (N-H) creep, the creep rate is given by [5]

$$(d\epsilon/dt)_{\rm NH} = \alpha_{\rm NH} (D_{\rm SD}/d^2) (\sigma \Omega/kT)$$
(2)

where $(d\epsilon/dt)_{NH}$ is the strain rate due to Nabarro-Herring creep, D_{SD} is the lattice diffusion coefficient, d is the grain size, σ is the applied stress, Ω is the atomic volume, k is Boltzmann's constant (1.38x10⁻²³ atom⁻¹k⁻¹), T is temperature and α_{NH} is a geometrical constant.

The form of equation (2) is fully consistent with the power law relationship (Eqn.1). Inspection of equation (2) shows, that when creep occurs by stress-directed flow of vacancies through the crystal lattice, $(d\epsilon/dt)_{ss}$ varies directly with the applied stress, σ . i.e. in terms of the power law relationship, n = 1. Furthermore, the dependence of $(d\epsilon/dt)_{ss}$ on temperature is governed largely by D_{SD} since, from equation (2)

Also, from diffusion we know the self-diffusion coefficient, D_{SD}, can then be expressed as [6]:

$$D_{SD} = D_0 \exp(-Q_{SD}/RT)$$
(3)

where D_0 is a constant and Q_{SD} is the activation energy for self-diffusion. Hence, when the power-law approaches are used to describe this behavior, n = 1 and $Q = Q_{SD}$. The Nabarro-Herring creep mechanism is dominant at higher temperatures and higher stresses.

(b) Coble creep

The other main type of diffusion creep mechanism is known as Coble creep. Coble creep involves a vancancy transfer along the grain boundaries (See Figure 2.9). This describes the transfer of vacancies only along a narrow zone adjacent to the grain boundary. Let the width of this narrow zone be δ . The area of the grain boundary zone intersecting a unit area of a polycrystal of average grain diameter, d, is given by (δ/d) . However, diffusion along this narrow zone is not determined by self-diffusion but grain boundary diffusion.



Figure 2.9 Schematic of Coble creep on an ideal grain, after [7].

For Coble creep, the creep rate is given by [5]

$$(d\epsilon/dt)_{\rm C} = 8(D_{\rm GR}/d^3)(\delta\sigma\Omega/kT)$$
(4)

where $(d\epsilon/dt)_C$ is the strain rate due to Coble creep, D_{GB} is the grain boundary diffusion coefficient, d is the grain size, σ is the applied stress, Ω is the atomic volume, k is Boltzmann's constant, T is the temperature, and δ is the grain boundary thickness.

Because the diffusion is determined by the grain boundary diffusion and not the lattice self-diffusion, the activation energy for Coble creep Q_C is less than the activation energy for self-diffusion. Since $Q_{SD} > Q_C$ we determine that Coble creep is the dominant creep mechanism at low temperatures (0.4 T_m) carried out at low stresses.

2.2.2. Dislocation controlled creep mechanisms

Dislocation studies using the transmission electron microscope (TEM) have shown that the creep process leads to changes in the dislocation arrangements in crystals. The undeformed crystal will originally have a uniform (low) dislocation density. But after creep deformation the dislocations are converted into very homogeneous structures. Some have a high density of dislocations (sub grain boundaries, etc.) and others are relatively dislocation free. There are four main types of dislocation controlled creep
mechanisms: dislocation glide, dislocation climb, glide limited by solute atoms, and grain boundary sliding. Deformation-induced twinning (due to coordinated dislocation movement) is rare in creep conditions, but it is observed in ordered materials.

(a) Dislocation glide

Dislocation glide is described as a dislocation moving on it's glide plane under the influence of large stresses.

The constitutive equation for creep by dislocation glide is given by [8]

$$(d\varepsilon/dt)_{DG} = (d\varepsilon/dt)_{o} exp(-U_{o}/kT) exp(\tau ba_{s}/kT)$$
(5)

where $(d\epsilon/dt)_{DG}$ is the strain rate due to dislocation glide, $(d\epsilon/dt)_0$ is a reference strain rate, U_0 is the activation energy for dislocation glide, τ is the applied shear stress, b is the Burger's vector, a_s is a fraction of the glide plane area, k is Boltzmann's constant and T is the temperature. Note: $ba_s = V^*$ is applied later. Also, it is important to note, in this mechanism diffusion does not play a controlling role.

(b) Dislocation climb

During deformation a material is considered to contain a number of dislocation sources. During creep these sources emit dislocations which move across their glide plane until the leading dislocations eventually become held up at an obstacle, forming a pile up array. This is thought of as a hardening process. Finally the leading dislocation will climb out of it's slip plane and continue on a parallel plane (See Figure 2.10).



Figure 2.10 Schematic of the dislocation climb mechanism, after [9].

The constitutive equation for creep by dislocation climb is given by [4]:

$$(d\epsilon/dt)_{DC} = (\alpha_{DC}DbG/kT(\sigma/G)^5)$$
 (6)

where $(d\epsilon/dt)_{DC}$ is the strain rate due to dislocation climb, α_{DC} is a geometrical constant, D_L is the lattice diffusion coefficient, k is Boltzmann's constant and T is the temperature.

(c) Dislocation glide limited by solute atmospheres

When alloying a pure metal a solute atom is introduced into the host matrix. The solute atom improves the creep resistance of the host material by impeding dislocation glide and recovery processes. However, when the stress exponent, n, decreases and is accompanied by a change from normal to inverse primary creep curve shapes, this is the symptoms of *solute atoms drag*. This behavior can be described as the solute atoms lattice strain field forms an atmosphere that encompasses the area around the dislocation. The solute/dislocation interaction is favored by large size differences between the solute and solvent atoms. The creep rate is then controlled by the rate at which the dislocations can move dragging their solute atom atmosphere. The creep rate is given by [5]:

 $(d\epsilon/dt) = [(\pi(1-v)fT\overline{D})/(6e^2Cb^5G)]^*(\sigma/G)^3$ (7) where $e = (\Omega^*/\Omega_b)^{1/3} - 1$ and $\overline{D} = (C_bD^* + CD_b^*)(1 + \ln a/\ln G)$, D =Chemical diffusion of solute atom, $D^* =$ Chemical dependent diffusion coeff. $\Omega^* =$ effective atomic volume of solute atom.

(d) Grain-boundary sliding

At elevated temperatures the grains in polycrystalline metals are able to move relative to each other. Grain-boundary sliding is a shear process which occurs in the direction of the grain boundary. It is promoted by increasing the temperature and/or decreasing the strain rate. Although most investigations indicate that sliding occurs along the grain boundary as a bulk movement of the two grains. Other observations indicate that flow occurs in a softened area a finite distance away from the grain boundary. Grain boundary sliding occurs discontinuously with time, and the amount of shear displacement is not uniform along the grain boundary. The amount of strain in the system is due to slip within the grains and grain boundary sliding. So, there is a close relation between the crystallographic slip and grain-boundary sliding.

Another way of accommodating grain-boundary strain at high-temperatures is by the formation of folds at the end of a grain boundary. Another recovery process is grain-boundary migration, in which the grain boundary moves normal to itself under the influence of shear stress and relieves the stress concentrations.

(e) Deformation-induced twinning

Mechanical twinning is a mode of plastic deformation that occurs by shear as the result of applied stresses. The shear associated with mechanical twinning is uniformly distributed over an entire deformed volume rather than localized on the discrete slip planes. The movement of the atoms is only a fraction of an interatomic spacing relative to the atoms in the adjacent plane. There is no change in crystal structure but a reorientation of the crystal lattice. After mechanical twinning, the deformed portion of the crystal becomes mirror symmetric with the undeformed matrix (See Figure 2.11).



Figure 2.11 Definition of twinning, [10].



A model used to describe a twin can be seen in Figure 2.12.



Where K1: twinning plane (the first undistorted plane).

K2: conjugate twinning plane (the second undistorted plane).

n1: twinning direction (the shear direction).

n2: conjugate direction (the intersection of the plane of shear with the second undistorted plane).

s: normal to the plane of shear.

2.3. CREEP EQUATIONS

During creep deformation several deformation mechanisms may be acting simultaneously, or different deformation mechanisms could be acting at different creep regimes. So, this may cause large differences in the values of the creep response. Most of the creep deformation equations describe the steady-state creep diffusion controlled mechanisms. However, some equations describe dislocation controlled mechanisms and power law breakdown, depending on the creep area of interest.

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2.3.1 Power law equation

One of the equations that is commonly used for creep is the power-law equation. The power law representation of high temperature creep is defined by the equation below[2]:

$$(d\varepsilon/dt)_{ss} = A\sigma^{n} exp(-Q/RT)$$
(1)

2.3.2. Mukerjee-Dorn equation

There are many adaptations to the power-law to "fine tune" the equation to be more specific and to have a greater accuracy. One of these power-law related equations that is used for creep behavior is the Mukerjee-Dorn (MD) equation[12]:

$$(d\varepsilon/dt)_{ss} = (AD_0Gb/kT)(\sigma/G)^n(b/d)^p exp(-Q/RT)$$
(8)

where T is the temperature, k is Boltzmann's constant, R is the gas constant, d is the grain size, p is the grain size exponent, b is the burgers vector and A is a geometric constant

The MD equation has an obvious relation to the power-law equation, but it takes the grain size and the temperate dependence of the elastic modulus. Comparing the two equations the differences are, a $(b/d)^p$ grain size ratio parameter, an inverse temperature factor and the temperature dependence of G is in the MBD equation. This refinement provides a structure that is useful for exploring mechanistic details. i.e. when plotting the creep data at different strain rates and at different temperatures, the plot should fall on the same line if the same deformation mechanism is responsible.

2.3.3. Power law breakdown

As mention previously, the steady-state creep condition is the most popular creep regime to determine experimental data. However, some research is involved in other creep conditions such as power law breakdown. During power law breakdown the Sellers-Teggart [12] equation is used to describe the creep behavior:

$$(d\varepsilon/dt) = AD/\alpha^{n}b^{2*}(\sinh\alpha(\sigma/E))^{n}$$
(9)

where $\alpha = (\sigma/E)^{-1}$

This equation describes the creep conditions when the stresses are very high, and powerlaw creep does not apply.

2.3.4. Composite modeling

To model the creep behavior at intermediate temperatures and stresses, Raj and Langdon [13] have reported that mechanisms controlling the creep behavior at intermediate temperatures are non-diffusional. By exploring a number of different possibilities, they found that the most important type of deformation mechanism was the Obstacle-limited glide of dislocations in the subgrain interior. Nix and Ilschner account for the smooth transition from power-law creep with increasing stress and decreasing temperature, by assuming that deformation mechanisms in the *hard* region occurs by diffusion controlled recovery and in the *soft* region by thermally activated obstacle-limited dislocation glide, creep rate. The following equation was given[13]:

$$\boldsymbol{\varepsilon} = \boldsymbol{\varepsilon}_{s} + \boldsymbol{\varepsilon}_{h} \tag{10}$$

Others have applied these ideas to specific materials, i.e. Soyboyeo [8] and Hazeldine [14]. The soft region (thermally activated obstacle-limited dislocation glide) [12]:

$$\varepsilon_{\rm s} = 0.5 \times 10^{12} (\sigma/G)^2 \exp[-(0.5 k_{\rm f} G_0 b^2 d_{\rm oc} - (\sigma - \sigma_{\rm b}) V^*)]/RT \quad (11)$$

where k_f = obstacle strength, G_o = shear modulus at 0 K, V* = activation volume, d_{oc} = stacking fault width at zero stress, σ_b = ave. long range back stress,

The hard region is determined by diffusion controlled recovery[13]:

$$\varepsilon_{\rm h} = 3000 (D_{\rm SD} {\rm Gb/kT}) (\sigma/{\rm G})^4 \tag{12}$$

where D_{SD} = lattice diffusion coefficient

The activation energy for obstacle-controlled glide, $Q_g[13]$:

$$Q_g = 0.5k_f G_0 b^2 d_{oc} - (\sigma - \sigma_b) V^*$$
(13)

The magnitude of Q_g at zero stress is as predicted $Q_g = 290$ kJ/mol at $\sigma = 0$, $k_f = 0.2$, $d_{oc} = 6b$

for a weak obstacle, assuming $\sigma_b = 0.5\sigma$

CHAPTER THREE

TITANIUM ALUMINIDE TIAI

3.1. CRYSTAL LATTICE STRUCTURES

In this section some basic understanding of the two crystal lattice structures of $L1_0$ (fct) for the gamma (TiAl) and the DO₁₉ (hcp) for the alpha2 (Ti₃Al) was summarized.

3.1.1. L1₀

The gamma phase in TiAl has a $L1_0$ type lattice crystal structure where the titanium and aluminum atoms alternately stack in (002) planes (Figure 3.1). The $L1_0$ structure is a slightly tetragonally distorted, face-centered cubic with a temperature dependent composition range of 49 to 66 (at%) of Al. The ratio between the lattice parameters and in the direction of the tetragonal axis is c/a = 1.02. However, this value increases up to c/a = 1.03 with increasing Al content and decreases to c/a = 1.01 with decreasing Al content [15]. The γ -TiAl phase remains ordered up to it's melting point of about 1450°C. Because the c/a ratio is always close to unity, it is very close to the fcc crystal structure. However, there are more possibilities for complicated slip in $L1_0$ due to the anisotropic crystal structure. It is of interest to note that twinning deformation is relatively common in the gamma phase.



Figure 3.1 Lattice structure of L1₀, after [16]

3.1.2. DO₁₉

Because TiAl materials typically have 1-10 volume% of the alpha phase, which has a DO_{19} type lattice crystal structure, it is of interest. The DO_{19} structure is based off the hexagonal closed-packed structure, but the a coordinate basel plane axis has been replaced by a value of twice as large (2a) to accommodate the lattice structure. It's relation to the hcp crystal lattice can be seen on the next page in Figure 3.2. The structure is based on the assumption of having a stoichiometric Ti₃Al. However, the system is often off-stoichiometric with Al atoms in Ti sites. Therefore, this causes a slightly disordered lattice. Also, it is interesting to note that according to Yamaguchi [16] deformation twinning is extremely rare in DO_{19} structures.



Figure 3.2 Lattice structure of DO₁₉, [16]

3.1.3. Differences between $L1_0$ and DO_{19}

The main difference between the $L1_0$ and the DO_{19} is the stacking sequence. The stacking sequence for the $L1_0$ (gamma) is ABCABC, while the DO_{19} (alpha2) stacking sequence is ABABAB. So the interface between gamma and alpha2, as in the lamellae of TiAl has a stacking sequence that is ABCABC:ABABAB. This interface corresponds to the (111) plane of the $L1_0$ and the (0001) basel plane of the DO_{19} . However, three <1120> directions on the (0001) basel plane of the hcp alpha phase are all equivalent while the [110] direction and other two <011] directions on the (111) plane are not equivalent to each other. Thus, the gamma has six possible orientations of [110] on (111) in the gamma phase with respect to <1120> on (0001) in the alpha phase.

3.2. MICROSTRUCTURE

Extensive progress and improvements in two-phase TiAl alloys have been made in the last five years. This is due to the fact that TiAl has become a candidate for high-temperature light-weight structural applications. The TiAl phase possesses a wide composition range and it extends primarily on the Al-rich side. While the TiAl compounds with Ti-rich compositions exhibit a two-phase microstructure are composed of the γ (TiAl) phase and a small amount of α_2 (Ti₃Al) phase. The recent interest in TiAl compounds has been primarily devoted to the two-phase γ/α_2 alloys rather than the Al-rich TiAl compounds with a single-phase γ structure. This is because the two-phase material is more ductile and tougher than the single phase compound.

In the last few years, improvements in the mechanical properties of the two-phase alloys has been accomplished. Most of the work was directed toward improving the tensile ductility; for example, two-phase alloys with tensile elongation as high as 4% [17] have been developed through alloying with ternary elements and controlling their microstructure by thermomechanical processing. However, these improvements in room-temperature ductility have been traded for other mechanical properties such as toughness and high-temperature strength. This part of the paper will review recent advances in our understanding of microstructure and its relation to the physical properties of two-phase TiAl alloys.

The phase equilibria and transformations in the near-equiatomic region of the TiAl diagram have been investigated, and it has been concluded that the beta+gamma two-phase region does not exist but instead, the alpha+gamma two-phase field extends up

to the peritectic temperature, as seen in the phase diagram proposed by McCullough et al. [18](See Figure 3.3). So, the solidification in near-equiatomic compositions proceeds along the two peritectic reactions $L + \beta \rightarrow \alpha$ and $L + \alpha \rightarrow \gamma$, or the single peritectic $L + \alpha$ $\rightarrow \gamma$ depending on the cooling rate. The phase diagram of Figure 3.3 indicates that the α phase decomposes into the α_2 and γ phases through a eutectoid reaction (at 1398 °C). However, some references[19] have suggested that it is impossible to observe the eutectoid transformation due to the fact that there is a substantial difference in the ease of nucleation between the α_2 and γ phases. The gamma precipitate is more sluggish than the α/α_2 transformation and thus the gamma phase can precipitate out of either α or α_2 phases depending on the cooling rate.



Figure 3.3 TiAl phase diagram around the gamma phase, [18]

e.

One of the most interesting features of the phase diagram of Figure 3.3, is the fact that the alpha single phase field extends all the way up to 1450°C in the near equiatomic region. That is, alloys with nearly equiatomic compositions can be heated into the single phase field where aluminum is completely in solution in the disordered hexagonal close-packed (hcp) structure of titanium and thus may be heat treated under a number of different conditions to develop a wide variety of mircostructures. This is analogous to high-carbon steels which can be heated in the austenite single-phase field and subsequently heat treated under appropriate conditions to develop the desired properties. Such a property has not been found in any other intermetallic compounds which have been studied as possible candidate for high-temperature structural applications. This uniqueness together with the exceptional combination of light weight, superior strength and oxidation resistance make the near-equiatomic TiAl alloys extremely attractive for high-temperature service in the aerospace field.

3.2.1. Lamellar structure

Most of the research covered in the literature discusses the lamellar structure. The lamellar structure is produced from two-phase TiAl alloys with near-equiatomic compositions. They are prepared by the usual ingot-metallurgy methods, where the gamma phase precipitates from the alpha phase producing a lamellar structure. The lamellar structure is composed of the transformed gamma and remaining alpha lamellae. Gamma lamellae are formed in such a way that closed-packed planes and directions in the gamma phase are parallel to the corresponding planes in directions in the alpha phase. In other words, the (0001) basel plane of the hcp alpha phase interfaces with the (111) plane of the tetragonal L10 structure for the gamma phase. According to Yamaguchi [20] the gamma phase can be formed in six possible orientations corresponding to the six possible orientations of [110] on (111) in the gamma phase with respect to $[11\overline{2}0]$ on (0001). The different crystal lattice structures will be discussed in a later section.

3.2.2. Thermomechanical structure

Most of the newer literature discusses the microstructures that are developed through thermomechanical processing. Ingots of two-phase TiAl alloys prepared by ingot metallurgy methods are usually HIPed, hot-worked, heat treated and cooled at an appropriate rate to obtain a specific microstructure and desired resulting mechanical properties. Such thermomechanical processing can provide a wide variety of microstructures in comparison to simple heat treatments without hot-working. The hot-working that is most common is usually isothermal forging, and is typically conducted near the eutectoid temperature. Such hot-working generally results in the structure consisting mostly of fine partially recrystallized gamma grains. Hot-working is then subjected to a heat treatment for further microstructural control. The characteristics of these microstructures have been cataloged by Kim and Dimiduk [21]. They classified the microstructures into four groups: Near-gamma (NG), Duplex (DP), Nearly lamellar (NL) and Fully lamellar (FL).

(a) The near-gamma (NG) structure is obtained by an annealing heat treatment at temperatures just above the eutectoid temperature and is characterized by coarse gamma grains and banded regions consisting of fine gamma and alpha2 grains. This type of microstructure is also known as equiaxed γ . (b) The Duplex (DP) microstructure has a microstructure composed of fine gamma grains, and fine lamellar grains. This is obtained only by combination of hot-working and subsequent heat treatment at temperatures where the volume fractions of alpha and gamma phases are roughly equal. Competitive growth between the alpha and gamma phases during heat treatment results in the fine duplex microstructure.

(c) The nearly lamellar (NL) structure is composed of coarse lamellar grains and a small volume fraction of fine gamma grains. Nearly lamellar is usually obtained by heat treatment of as-cast ingots.

(d) The fully lamellar (FL) is the same as that observed in as-cast conditions. They are obtained by heat treatments at temperatures a little below and above the alpha transus line.

A comparison of the creep resistance of duplex, single phase γ and fully lamellar microstructures tested under the same stress and temperatures can be seen in Fig-

ure 3.4.



Figure 3.4 Creep resistance comparison of different microstructures, [22].

The effects of alloy element additions to the two-phase material is a very complicated issue. The literature on the subject of alloying is concerned with the effects alloying has on the microstructure and consequently on the mechanical properties. These alloy elements additions and their reported effects are listed in the table below [19]:

Table 3.1Alloying effects observed in gamma-based alloys

Elements	Reported effects
Al	It strongly affects ductility by changing the microstructure. Best ductility occurs in the range of 46-50 at%.
В	Additions of >0.5 at% refine grain size, and improve strength and workability. Doping with B generally increases castability.
С	Carbon-doping increase creep resistance and reduces ductility.
Ст	Additions of 1 - 3 at% increase the ductility of duplex alloys.
	Additions of >2 at% enhance the workability and superplasticity. Additions of >8 at% greatly improve the oxidation resistance.
Er	Its additions change the deformation substructures and increase the ductility of single-phase gamma.
Fe	The addition of Fe increases fluidity, but also the susceptibility to hot cracking.
Mn	The addition of 1 - 3 at% Mn increases the ductility of duplex alloys.
Мо	The addition of Mo improves the ductility and strength of a fine- grained material. It also improves the oxidation resistance.
Ni	Increases fluidity.
Nb	The addition of Nb greatly improves the oxidation resistance. It slightly improves the creep resistance.



Table 3.1 (cont'd)Alloying effects observed in gamma-based alloys

<u>Elements</u>	Reported effects
Р	Doping with P decreases the oxidation rate.
Si	An addition of 0.5 - 1 at% Si improves the creep resistance. The addition of Si also improves the oxidation resistance. The addition of Si increases fluidity, but reduces the susceptibility to hot cracking.
Ta	The addition of Ta tends to improve the oxidation and creep resistance. It increases the susceptibility to hot cracking.
v	The addition of 1 - 3 at% V increases the ductility of duplex alloys. Its addition generally reduces the oxidation resistance.
W	The addition of W greatly improves the oxidation resistance. It improves the creep resistance.

(a) Ti-48Al-2Cr-2Nb alloy

The duplex alloy, Ti-48Al-2Cr-2Nb (at%) is one of the two-phase alloys which have been a subject of many studies [8,15,19,23,24,25]. This alloy is reported to have attractively balanced mechanical properties. The addition of Cr in near-gamma twophased alloys in duplex form shows good ductility at room temperature because of an increased volume fraction of equiaxed soft gamma grains. This is obtained by the Cr depressing the alpha-transus, thereby raising the Al content of primary alpha phase and leading to a reduction in volume fraction and width in the alpha2 lamellae. The addition of Nb has been reported to improve the oxidation resistance of the two-phase alloys, but does not seem to be beneficial to the ductility of the two-phase alloys. The effect of the addition of Nb together with Cr on the mechanical properties of the two-phase alloys needs to be clarified. Of Nb or Cr, in particular, Nb is considerably enriched in the alpha2 phase, so

the mechanical properties of the alpha2 phase and therefore the lamellar grains would be changed [19]. However, further studies on this subject are needed.

Another important aspect of the microstructure is the effect of the interstitial impurities. The nominal oxygen content in γ -based materials are typically 500 - 1000 ppm [19]. However, the actual content is lower in the γ -phase of the duplex structure, since the α_2 phase tends to absorb oxygen. The α_2 also tends to absorb nitrogen and carbon impurities. The impurities tend to increase strength and reduce ductility. Additionally, they may have beneficial effect of reducing the creep rate.



CHAPTER FOUR

LITERATURE SURVEY OF CREEP IN TIAI

4.1. INTRODUCTION

For the last decade, intermetallic alloys based on γ -TiAl have become promising candidates for high temperature service. Surprisingly, the issue of creep in near γ -TiAl based alloys has been addressed only marginally and, in contrast to the large amount of literature on microstructure and mechanical properties, a very limited number of publications have been concerned with the creep properties of these alloys. Studies of some of the microstructures and their resulting creep deformation mechanisms are still missing.

The creep deformation mechanism(s) for TiAl appear to be very complicated. The results obtained from this literature survey show the creep deformation is a function of the composition of the specimen, grain size, thermomechanical processing (microstructure), as well as the applied stress and test temperature. Because there is so much various data to catalog, the conditions of the different tests and trends in the data are listed. However, these trends may be limited to a single composition, microstructure or test condition.

Again, little is known about the creep behavior of this material. This is partially due to the fact that most of the creep experimental results are not exhaustively analyzed so the creep deformation mechanisms are only partially understood. Also, the values obtained to describe the creep characteristics for different test conditions have a

large variance, and these large differences in values are not well understood. These large differences in the creep parameters of gamma-based TiAl will be investigated in an attempt to understand which possible deformation mechanisms control creep.

4.1.1 Trade-off approach

There are many different compositions and microstructures of titanium aluminides. Each composition affects mechanical properties and processability through microstructural control. The most important aspect of the composition is the amount of aluminum present. Duplex alloys containing 45-50 at% Al are generally most desirable. Alloying additions that improve the ductility, oxidation, creep resistance, and fabricability have been identified in Table 1, but those additions may adversely affect other properties at the same time. Therefore, it is important to empirically determine the trade-off of each alloying approach. The selection of the alloying approach also depends on the application, which defines the property requirements and the preferred processing route. Based on the progress and improvements, several engineering two-phase alloys in duplex microstructure form with 3 - 4% room-temperature ductility and improved strength have been developed. Creep strength has also been markedly improved in the last few years.

4.1.2. Comparison of different microstructures creep resistance

The creep resistance of each different composition and microstructure are different. The fully lamellar (FL) structure is not only tougher, but more resistant to creep than the single phase (NG) or the duplex (DP) structure. The fully lamellar dramatically reduces the initial transient creep and the rate of steady-state creep(See Figure 3.4). As a

result, the time to creep to 0.2% strain increases by two orders of magnitude. However, the fully lamellar structure is also the most brittle at room temperature. Therefore, it makes the fully lamellar structure hard to machine and the fabrication of useful high temperature components very expensive.

The duplex structure has moderate creep resistance but is the best trade-off of creep resistance for ductility. The composition of Ti-48Al-2Cr-2Nb is the alloyed composition that seems to have the best of both worlds. A good creep resistance with up to 3-4% ductility. However, this composition also has a small grain size which effects the creep properties.

4.2. PROBLEMS IN UNDERSTANDING CREEP OF TIAI

There are many problems in determining which creep deformation mechanisms are responsible for the steady-state creep behavior of TiAl. The following will summarize the most obvious problems which are: 1) minimum creep rate with no steady state deformation, 2) no instantaneous strain or primary creep in some compositions and 3) dynamic recrystallization. However, there are many more subtle problems in understanding of creep of TiAl that are not as apparent as the ones that will be discussed.

4.2.1. Minimum creep rate

The most obvious problem is the fact that TiAl does not have a steadystate creep rate regime. The term steady-state assumes the strain rate to be a constant, i.e. when the recovery rate balances perfectly with the strain hardening rate for a significant fraction of the creep life. However, the strain rate reported for a polycrystalline TiAl in the duplex condition exhibits a minimum strain rate followed by an increase. Hence no steady-state strain rate is truly observed, though the minimum provides a basis for comparison. Some examples of experimental creep curves are given in Figure 4.1 [24].



Figure 4.1 Minimum creep rate of near gamma-TiAl, [24]

4.2.2. No instantaneous strain

Oikawa [26,27,28,29,30] reports that under low stresses, no instantaneous strain can be observed in equiaxed gamma (Ti-53Al). The strain at the primary stage is very limited and an apparent steady state soon appears. After this low strain rate stage, the creep rate increases up to a high value which is more than one order of magnitude higher than the steady state creep rate. This type of creep behavior is unusual because there is shift in deformation from a slower one to a faster one, depending of the amount of strain in the system. The mechanisms of this type of creep response have not yet been clarified.

4.2.3. Dynamic recrystallization

Another problem that obscures our understanding of creep in TiAl is the dynamic recrystallization (DRX) that occurs. The strain rate passes through a minimum and increases to a steady state value. The characteristic steady state associated with dynamic recovery exhibits a lower activation energy (269 kJ/mol) than the apparent steady-state stress during DRX (400 kJ/mol)[25]. The difference between the two activation energies is about 15% higher for the DRX than the activation energy for the steady-state recovery. At larger strains the creep conditions are dominated by DRX. There is a tendency for the recrystallized grains to nucleate along prior grain boundaries of the original microstructures[25]. DRX has been reported to be important at higher-temperatures (approx. 760°C) and at strains of approximately 20% strain. The physical impact of the creep characteristics by DRX could be one reason why the values for the activation energy have such a large variance.

4.3. DEFORMATION HISTORY INDEPENDENCE

Some of the studies in the literature [8,15,19,22,23,24,25] assume the creep specimen is deformation history independent. That is, the creep deformation taking place at σ_3 , is not influenced by prior creep deformation which occurred at σ_2 and σ_1 previously. In other words, a fresh creep specimen loaded initially at a stress level equal to σ_3 and a specimen that was initially loaded at a different stress then, after a stress change, was loaded to σ_3 , at the same temperature, would creep at identical or nearly identical rates. Therefore, the same specimen is used to determine the creep behavior at different stress regimes. This type of approach saves time and money. This technique is also known as the

stress increment technique.

4.4. DIFFUSION CONTROLLED CREEP IN TIAI

As mentioned previously, diffusion controlled creep can be classified as either Nabarro-Herring creep, where $Q = Q_{SD}$, or Coble creep, where $Q = 1/2 Q_{SD}$, depending on the temperature and stress regime examined. However, both creep models are based on self-diffusion.

4.4.1. Self diffusion of Ti-54Al

The literature has shown a variety of values for the activation energy, ranging from 80 to 600 (kJ/mol) [26,27,28,29,30,31] depending on the different conditions of each creep study. But a majority of the literature suggest a value near 291 kJ/mol, the value reported by Kroll, Mehrer for Ti diffusion[32]. The value of 291 kJ/mol was obtained by a concentration-depth profile of the radioisotope Ti⁴⁴ in binary Ti-54Al using a serial sectioning technique. Although much work has been done on other intermetallic structures, such as the B₂, little or no work has been reported on the diffusion in the L10 type crystal structure. Kroll and Mehrer where one of the first to investigate the self-diffusion of the intermetallic γ -TiAl. The figure below shows the arrhenius relationship for γ -TiAl for the self-diffusion of Ti(See Figure 4.2).



Figure 4.2 Plot of self-diffusion of Ti-54Al, [32]

Using the equation for diffusion one can obtain a value for the activation energy equation (3).

$$D = D_0 \exp(-Q/RT)$$
 [6] where $D_0 = 1.53 \times 10^{-4} \text{ m}^2/\text{s}$ and $Q = 291 \text{ kJ/mol}$

Because the compound γ -TiAl does not have a congruent melting point but instead decomposes by a peritetic reaction into β -TiAl and a melt (See Figure 3.3), Kroll and Mehrer instead use the value of the peritetic reaction in place of the melting temperature, T_m. Following this argument, Kroll and Mehrer estimate a value of 261 kJ/mol instead of the measured value of 291 kJ/mol. However, since their specimen composition is on the Al-rich side (Ti-54Al) of the phase diagram, which may have a significant impact on the physical properties, it is not clear if the diffusion mechanism is the same in the near- γ range. Also, Ti-54Al (NG) has a microstructure that is gamma without much alpha2 present. Therefore, the value may not be directly applicable for the lamellar-based on microstructural constraints.

The value reported for the self-diffusion is very important for the mathematical modeling of creep. Because, this value will help to pinpoint the rate-limiting deformation mechanisms for different creep regimes. However, the deformation mechanism(s) are hard to determine due to the large variance in the values reported by the literature for the activation energy.

4.4.2. Alloys containing chromium

The activation energies determined from creep experiments on single phase alloys are much higher than the interdiffusion (of Ti and Al) activation energies of 80 and 168 kJ/mol determined by Ouchi from diffusion couples of α_2 and γ alloys, respectively [54]. This suggests that factors other than diffusion play a significant role in creep deformation of gamma alloys. These may include the dislocation/cell structures, and pipe diffusion phenomena. Alternatively, very high creep activation energies in the Cr-containing alloys may be due to the strong effects of Cr on the diffusion kinetics in gamma alloys. Cr has been shown to slow down the transformation kinetics in duplex gamma alloys, but it's effects on diffusion have not been studied. Nevertheless, it is clear from studies of phase transformations in gamma alloys that Cr reduces the diffusion kinetics by 1-2 orders of magnitude [8].

The investigation of the phenomena of the lower diffusion rate associated with the creep deformation of Cr-containing gamma alloys, unfortunately must rely on data obtained for non-chromium containing gamma alloys. Nevertheless, although the available single and multi-phase gamma diffusion data are insufficient for rigorous analysis, they do provide the basis for preliminary analysis of the trends in creep behavior in the low stress regime.

4.5 DISLOCATION CONTROLLED CREEP IN TIAI

It is well known that at higher stresses, dislocation motion will contribute more to deformation than diffusion. This has led to dislocation studies using the transmission electron microscope (TEM), that have shown that the creep process leads to changes in the dislocation arrangements in crystals. The two types of dislocation controlled creep mechanisms that will be discussed are twinning and dislocation glide-climb.

4.5.1. Twinning

To most material scientists, mechanical twinning is considered to occur only in crystalline materials at high strain rates and for low temperature conditions. However, recently several studies investigating TiAl have reported that mechanical twinning occurs at creep conditions with low strain rates and at intermediate to high temperatures.

Since the introduction of the twinning type of deformation mechanism in TiAl, it's role in the creep process has been largely overlooked. Jin and Bieler [15,33,34] have shown that twinning occurs in various ways, including pseudo-twinning in different parts of the microstructure. In equiaxed gamma grains, twins nucleate from grain boundaries due to stress concentrations at grain boundaries and triple points. While twinning is not supposed to occur in the alpha2, the lamellar structure also includes some gamma that have shown cross-twinning. Twinning occurs at certain temperatures, while twinning does not occur at other temperatures. This type of temperature sensitive behavior has been indicated in an analysis of the ductile to brittle transition [19]. It has been observed that twinning has a significant role in the transition, by causing the material to be more brittle than before twinning. A description by Shih [23] found twinning in the lamellar (FL) type structure at a creep temperature of 760°C, while Loiseau [35] also found twinning in

Ti-54Al in single phase gamma (NG) type structure at a creep temperature of 800°C. However, Huang and Kim [36] did not find twinning in a two phase alloy at a creep temperature of 900°C. Clearly, twinning is an important deformation mechanism in the range of creep rates and temperature. A representation of the twinning system for the gammabased TiAl can be seen in Figure 4.3.



Figure 4.3 Representation of twinning system, [34]

The twinning elements shown in Figure 4.3 are listed as relative orienta-

tions as viewed along [110] direction. The dashed line in Figure 4.3 indicates the position

of K_2 after true-twinning.

Table 4.1Mechanical twinning elements of the thin twin layer

К1	К ₂	η_1	η2	Shear	
(111)	(111)	[11 2]	[112]	0.707	

In order to better understand the relation between the twin elements, and

their orientation to tensile axis of the specimen Figure 4.4 provides a reference.



Figure 4.4 Twin orientations as viewed along the <110> direction, [15]

The mechanical twinning mechanisms is based on the dissociation of normal dislocation b = 1/2[110] into two partial dislocations[33,34]:

$$1/2[110] \rightarrow 1/3[111] + 1/6[11\overline{2}]$$

This leads to the glide of a/6[112] partials that cause deformation-induced twinning in γ alloys. However, there been no reports of models describing how creep by deformation-induced twinning contributes to creep deformation of γ -based titanium aluminides. Soboyejo and others [8] report that the simple twin model neglects additional strains that are induced by coordinated movements of the partials. The effects of shear strains that result from deformation-induced twinning have been neglected due to the complexity of the modeling efforts required for the assessment of such strain distributions.

4.5.2. Dislocation glide-climb

At higher stresses, dislocation motion will contribute more to deformation than diffusion. However, a stress may be high enough for a dislocation to move but not sufficient to overcome obstacles. Diffusion assisted dislocation motion is achieved allows the dislocation to climb around obstacles so they can continue to slip on another plane. and thus permit continued deformation. Haves and London [37] have reported an activation energy of Q = 326.4 kJ/mol and a stress exponent n = 4.95 for the composition Ti-48Al-1Nb y TiAl. The value reported in the study for the stress exponent is in agreement with dislocation climb controlled power law creep. Hayes indicates twinning occurs at low SFE, while climb occurs at higher stresses and high SFE [38]. A study of the combination of these two types of dislocation mechanisms acting either simultaneously or independently is needed to understand their role in the creep process. According to the incoherent twin boundary structure the true-twin plane energy can be estimated to be about one-half of the intrinsic stacking fault energy or extrinsic stacking fault energy in TiAl, assuming that the intrinsic stacking fault energy and the extrinsic are equal to each other. Based on the intrinsic stacking fault energy reported in the literature [15], 70 mJ/m². the true twin plane energy in TiAl is about 35 mJ/m^2 .

4.6.3. Geometrical constant A

An investigation into the power-law equation (Eqn. 1)[2] has yielded many interesting questions. The meaning of the "A" term is considered next.

$$(d\varepsilon/dt)_{ss} = A\sigma^{n} exp(-Q/RT)$$
(1)

For a vast majority of the creep data in the literature, "A" is ignored. However, Sherby and

Burke [39] reports that the A value is a structure dependent constant which reflects the influence of stacking fault energy (SFE). It is considered that low values of A correspond to materials having a low SFE [39]. The SFE is also believed to be a function of the aluminum content, where the stacking fault energy decreases with increasing aluminum content. A minimum exists in the stacking fault energy within the 48 to 51 (at%) aluminium range [37]. Martin and Lipsitt [40] observed a significant reduction in the steady-state creep rate of the TiAl alloy Ti-48.7Al-2.2W when the W was held in solid solution as opposed to previous creep rates measured for the same alloy with the W precipitated out on internal surfaces in the form of fine beta phase particles. The apparent stress exponents and activation energies for creep deformation were found to be nearly identical for the two conditions. It was concluded that the significant differences in creep resistance could not be explained on the basis of the stress or temperature dependence leaving the pre-exponential constant A as the only remaining variable. Table 4.5 shows the values of A obtained from previous studies, along with the apparent stress exponents and activation energies used in the calculations of A.

Table 4.2	
Comparison of different creep parameters and A	37

<u>Composition</u>	σ	Temp	<u>n</u>	Q	Α	REF
(at%)	(MPa)	(°C)	(1	cJ/mol)		
Ti-48.7Al-2.2W*	173 - 345	700 - 900	4.5	400	2.20 x 10 ⁵	[13]
Ti-48.7Al-2.2W	172.4	750 - 900	5.5	370	2.83 x 10 ⁷	[13]
Ti-50.3A l	206 - 241	700 - 850	4.0	300	1.78 x 10 ⁷	[26]
Ti-48Al-1Nb	103 - 241	704 - 800	4.95	326.4	6.34 x 10 ⁷	[37]
Ti-5 3Al-1Nb	69 - 103	832 - 900	6.0	560	3.00 x 10 ¹⁰	[37]
where * = W in so	lid solution	า				
Martin and Lipsitt pointed out that a difference in the pre-exponential constant A, could result in differing steady-state creep rates without influencing the stress or temperature dependence of the steady-state creep rate. Table 4.2 indicates that the basic deformation process remains unaltered, but occurs at different velocities. Therefore, it is indicated that lowering the SFE (lower A) reduces the climb velocity by making climb more difficult. If dislocation climb is the rate controlling mechanism, we expect an apparent creep activation energy close to that of self-diffusion and a stress exponent in the range of 4-5.

Hayes indicates that the A value, obtained by his method, varies from one study to another. One possible reason for these large differences in the A parameter could be explained by the changes in microstructure from one composition to the next. For example, the fully lamellar (FL) and the duplex (DP) structure, would have different A values. However, the results are preliminary and more work needs to be completed to understand how this pre-exponential A value effects the creep deformation process.

4.6. DEFORMATION MECHANISM TRANSITION

As mentioned previously, the creep deformation mechanism that is dominate in one temperature and stress regime is often replaced by another deformation mechanism at a different temperature and stress regime. To illustrate this deformation mechanism transition a list of the potential creep deformation at temperatures of $(T > 0.4T_m)$ for equiaxed gamma [43] is shown in table 4.3.

Table 4.3	
Creep deformation Mechanisms at $T > 0.4T_m$ [43]	3]

Temperature	stress	<u>n</u>	Q	p	<u>mechanism</u>
Low	Low	1	Q _{GB}	3	Coble
		1	Q _{SD}	0	Harper-Dorn
	Intermediate	7	Q _P	0	Climb-recovery
	High nor	linear	Q _{SD} ?	0	Power-law breakdown
High	Low	1	Q _{SD}	2	Nabarro-Herring
		1	Q _{SD}	0	Harper-Dorn
	Intermediate	5	Q _{SD}	0	Climb-recovery
		3	QI	0	Viscous glide
	High non	-linear	Q _{SD} ?	0	Power-law breakdown

where Q_{SD} = activation energy for lattice self diffusion

 Q_{GB} = activation energy for grain boundary diffusion (approx. = 1/2 Q_{SD})

 Q_P = activation energy for pipe diffusion

 Q_I = activation energy for interdiffusion of solute atoms

4.6.1. Unique creep behavior of Ti-48Al-2Cr-2Nb

A study of the steady-state creep properties by Wheeler and London [25] shows for the composition of Ti-48Al-2Nb-2Cr the steady-state strain rate has a unique power-law behavior, see equation 1. The results indicate the stress exponent, n, increases from n = 3 at low stresses, to n = 7 at higher stresses. See Figure 4.5 on the next page.



Figure 4.5 Creep behavior of Ti-48Al-2Cr-2Nb (DP), [25]

The stress exponents here are not characteristic of stress-assisted diffusion creep process, such as Nabarro-Herring or Coble creep, which typically have an exponent of n = 1, or dislocation creep processes which have an exponent of n = 5. However, Ruano and Sherby [39] have reported the stress exponent can be increased by two when pipe diffusion is the rate-controlling mechanism of creep deformation at intermediate temperatures. The power-law here can possibly be explained by creep being controlled by pipe diffusion at low stresses and then changing control to dislocation creep at high stresses. The mechanism can be thought of as being controlled by diffusion via dislocation alignment and subgrain formation during the creep deformation.

Wheeler [25] made an Arrhenius plot to indicate the activation energy and came up with a value of 300 kJ/mol which is in agreement with 291 kJ/mol found by Kroll. However, an increase in the activation energy greater than 400 kJ/mol appears at

high stress levels. This observation is consistent with the idea of a change in the controlling creep mechanism at higher stress levels, and it was correlated with DRX.

4.6.2. Aluminium content

As mentioned previously, the Al concentration is suggested to be related to the SFE. Therefore, the Al concentration is indicated to determine the steady-state creep rate. This report was confirmed by a study of equiaxed gamma (NG) with different amounts of aluminum by Oikawa [26,27,28,29,30]. Using the (MD) equation to analyze the data, a trend based on the aluminum content of different specimens, is in the table 4.4. With decreasing aluminum, the stress exponent, n, and the activation energy, Q increases. The conditions of the creep test was a compressive creep test at a temperature of 677- 927 ^oC and at stresses between 80 - 400 MPa.

Table 4.4
Relationship between aluminum content and creep parameters
at low temperatures and low stresses [26]

Composition (at%)	n	Q (kJ/mol)
Ti-53.4Al (NG)	4.0 - 4.5	330
Ti-51.5Al (NG)	4.5 - 5.5	330
Ti-50Al (NG)	5.0 - 5.7	380

This same change occurs in the other two compositions of lower aluminum content, Ti-51.1Al and Ti-50Al. Oikawa suggests that creep parameters such as the activation energy and the stress exponent are insensitive to microstructural change. This would imply that changes in the creep parameters are caused by the changing of deformation mechanisms from one type of mechanism to another. Also, Oikawa reports that the minimum creep rate depends on the creep condition. This adds another factor to the consideration of the creep rate. He notes the following trends associated with the aluminum content.

Table 4.5Relationship between different creep effects [29]

1. Concentration of Al decreases:

Grain size effect increases, the microstructural effect increases.

2. The effects Al concentration has on the properties decreases with:

Increasing stress and temperature, increasing grain size.

3. As the amount of strain increases:

Amount of twinning increases, amount of recrystallization increases.

Table 4.5 shows that the creep characteristics are changing due to the amount of aluminium in the specimen. Seertman [41] indicates a modification of the mathematical approach is needed to compensate for the effect. He states that in the steady-state condition the creep rate in two-phase alloys obeys the conventional power-law creep equation. The reported values of the apparent creep activation values are in the range of 300 - 600 kJ/mol and the stress exponent values are in the range of 4.0 - 6.0. The composition and grain size dependence of the minimum creep rate can be described by the follow-ing equation [41]:

$$d\mathcal{E}/dt_{ss-min} = A(1+X)^m d_0^{-p}$$
(13)

where A is a constant, $X = 2N_{AI}$ -1, N_{AI} is the mole fraction of Al, d_0 is the initial grain size and m is in the range of 10 - 20. The grain size exponent p is 3.7 - 4.7 for Ti - 50 at% Al and 0.5 - 2.3 for Ti - 51 at% Al. Again, the grain size effect on creep is found to be sensitive to the Al concentration, although the reason for this has yet to be established.

From Oikawa, the figures 4.6 and 4.7 show the change in deformation behavior of Ti-53.4 Al (NG)at a different stresses and temperature. It is not clear whether the deformation mechanisms changes from one type to another, or if two or more deformation mechanisms act simultaneously.



Figure 4.6 Transition of stress exponent of Ti-53.4Al (NG), [26,27,28,29,30]



Figure 4.7 Transition of activation energy of Ti-53.4Al (NG), [26,27,28,29,30]

4.7. COMPOSITE VIEW OF CREEP

Soboyejo and Lederich [8,42] report that the creep behavior of gammabased alloys with duplex alpha2 and gamma microstructure can be modeled by recognizing that the two phase alloys are composites that consists of alpha2 and gamma phases. However, these two phases are grouped differently into alpha2-gamma lamellar colonies and single gamma grains. The creep mechanisms in the individual constituents of the composite are thus assumed to be similar to those that would occur in polycrystalline alpha2 or gamma subjected to the same stresses and temperatures. This assumption neglects the different geometrical constraints and stress states that can exists at the boundaries between alpha2 and gamma grains. Nevertheless it does provide useful insights into the contributions of individual alpha2-gamma and gamma phases to the composite creep deformation process. The strain rate contributions from the alpha2-gamma and gamma phases are determined for the composite using a simple rule-of-mixtures approach, and constitutive equations for the assessment of diffusion-controlled and dislocation controlled creep.

Assuming that the equiaxed grains and lamellar colonies are arranged in series, i.e., constant stress conditions (See Figure 4.8), the diffusion coefficient of the composite can be estimated from a simple rule-of-mixtures to be:

$$D_{c} = F_{\alpha 2} D_{\alpha 2} + F_{\gamma} D_{\gamma}$$
(14)

where $D_c = Diffusion$ Coefficient, F = phase volume fraction of α_2 and γ phases respectively

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Figure 4.8 Idealized duplex grain, [8]

The value of the diffusion rate, D, is usually equivalent to the activation energy for diffusion-controlled creep deformation. Also, the experimental conditions of 200 MPa and 800°C and an observed creep rate of 6.44 x 10⁻⁷ were used to evaluate their model. Using the interdiffusion data of Ouchi [31], they obtained diffusion coefficients for single phase alpha2, $D_{\alpha 2}$ is $5.1 \times 10^{-8} \text{ m}^2 \text{s}^{-1}$ and for single phase gamma, D_{γ} is 1.2×10^{-6} $\text{m}^2 \text{s}^{-1}$. This gives composite diffusivity that is close to that of the γ phase. They used the following models to assemble a composite creep rate:

Diffusion-controlled creep may occur by the following two mechanisms:

For Nabarro-Herring (N-H) creep, the creep rate is given by

$$(d\epsilon/dt)_{\rm NH} = \alpha_{\rm NH} (D_{\rm L}/d^2) (\sigma \Omega/kT)$$
(4)

For Coble creep, the creep rate is given by

$$(d\varepsilon/dt)_{\rm C} = 8(D_{\rm GB}/d^2)(\delta\sigma\Omega/kT)$$
(5)

Dislocation-controlled creep may occur by the following two mechanisms:

For dislocation glide, the creep rate is given by

$$(d\varepsilon/dt)_{DG} = (d\varepsilon/dt)_{o} \exp(-U_{o}/kT) \exp(\tau ba_{s}/kT)$$
(6)

For dislocation climb, the equation is given by

$$(d\epsilon/dt)_{DC} = (\alpha_{DC}/h^{3.5})(D_L/M^{0.5})(\sigma\Omega/kT)$$
 (7)

where h = characteristic jog length associated with climb,

M = number of dislocation sources per unit volume.

The applied longitudinal stresses are applied to individual grains/laths which are idealized as cubes or cuboids. The different orientations of the grains or lamellar colonies with respect to the applied loads are also neglected, as well as possible interactions between creep mechanisms. The composite strain rate is thus given by

$$(d\epsilon/dt)_{c} = f_{\alpha 2}[(d\epsilon/dt)_{DC\alpha 2} + (d\epsilon/dt)_{DG\alpha 2} + (d\epsilon/dt)_{C\alpha 2} + (d\epsilon/dt)_{NH\alpha 2}] + f_{\gamma}[(d\epsilon/dt)_{DC\gamma} + (d\epsilon/dt)_{DG\gamma} + (d\epsilon/dt)_{C\gamma} + (d\epsilon/dt)_{NH\gamma}] +$$
(15)

where subscripts α_2 and γ denote the alpha2 and gamma phases respectively, and f is the phase volume fraction. The above equation includes a number of mechanisms contributing simultaneously to creep deformation in two-phase γ alloys.

The Soboyejo approach is to obtain values for each specific term in equation 15. The values are then compared to the observed strain rate. The report that Coble creep is the dominate creep deformation mechanism on the basis of the values obtained. The predicted composite creep rate for alpha2 and gamma using the coble creep equation is within one order of magnitude of the observed rate.

Soboyejo and others [8,42] have attempted to describe the microstructure of TiAl as a composite material. Hazzledine [14] also reports the lamellar microstructure as a composite that has a different model than Soboyejo. The values that were obtained for Soboyejo's model will be explored later in the chapter.

4.8. SUMMARY OF CREEP RATES

In order to gain a better understanding of some of the values for the creep rates reported in the literature, the following is a summary of the minimum steady-state creep rates obtained from a few studies in the literature survey. Table 4.6 lists the different compositions and microstructures of the TiAl studied, as well as the creep conditions for the creep rate obtained. The entire list of minimum steady-state creep rates obtained from the literature survey can be found in Appendix II.

Table 4.6
List of minimum steady-state creep rate and creep conditions

Composition (at%)	<u>T (°C)</u>	<u>o (MPa)</u>	(dɛ/dt) _{min}	<u>Ref</u>
Ti-48Al-1Nb	704	103.4	2.2 x 10 ⁻⁶	[37]
(DP)	704	206.8	5.7 x 10 ⁻⁵	
	704	241.3	1.2 x 10 ⁻⁴	
	75 0	103.4	1.3 x 10 ⁻⁵	
	75 0	206.8	3.3 x 10 ⁻⁴	
	75 0	241.3	1.0 x 10 ⁻³	
	800	103.4	7.2 x 10 ⁻⁵	
	800	172.4	5.9 x 10 ⁻⁴	
	800	206.8	2.4 x 10 ⁻³	
	85 0	103.4	4.0 x 10 ⁻⁴	
	850	172.4	5.0×10^{-3}	
	850	206.8	1.6 x 10 ⁻²	
Ti-50.3A1	700	103.4	2.0 x 10 ⁻⁶	[40]
(DP)	700	206.8	2.4 x 10 ⁻⁵	
	700	241.3	5.2 x 10 ⁻⁵	
	75 0	103.4	9.0 x 10 ⁻⁶	
	75 0	206.8	1.2 x 10 ⁻⁴	

Table 4.6 (cont'd) List of minimum steady-state creep rate and creep conditions

<u>Composition (at%)</u>	$T(^{o}C)$	<u>σ (MPa)</u>	(dɛ/dt) _{min}	Ref
Ti-50.3Al (cont'd)	750	241.3	3.0 x 10 ⁻⁴	[40]
	800	103.4	5.0 x 10 ⁻⁵	
	800	206.8	7.2 x 10 ⁻⁴	
	800	241.3	0.8 x 10 ⁻³	
	850	103.4	1.1 x 10 ⁻⁴	
	850	206.8	3.2 x 10 ⁻³	
	850	241.3	5.3 x 10 ⁻³	
Ti-48.7A1-2.2W	750	172.4	1.1 x 10 ⁻⁵	[40]
(DP)	800	172.4	4.0 x 10 ⁻⁵	
	850	137.9	4.8 x 10 ⁻⁵	
	850	172.4	2.1 x 10 ⁻⁴	
	850	241.3	6.8 x 10 ⁻⁴	
	850	310.3	4.1 x 10 ⁻³	
	900	172.4	9.2 x 10 ⁻⁴	
	95 0	172.4	5.5 x 10 ⁻³	
Ti-43Al	1000	35	1.0 x 10 ⁻⁵	[50]
(NG)	1100	400	2.0 x 10 ⁻²	
Ti-50Al	727	126	1.6 x 10 ⁻⁴	[50]
(NG)	927	398	8.3 x 10 ⁻³	
Ti-48Al-2Cr-2Nb (DP)	800	200	6.4 x 10 ⁻⁷	[8]
Ti-48Al-2Cr-2Nb	705	200	1.0 x 10 ⁻⁹	[25]
(DP)	705	300	1.0 x 10 ⁻⁸	
	760	200	1.0 x 10 ⁻⁸	
	760	300	6.0 x 10 ⁻⁸	
	815	200	6.0 x 10 ⁻⁸	
	815	300	6.0 x 10 ⁻⁷	

Table 4.6 (cont'd)
List of minimum steady-state creep rate and creep conditions

Composition (at%)	<u>T (°C)</u>	<u>σ (MPa)</u>	(de/dt) _{min}	Ref
Ti-53.4A1	900	100	1.0 x 10 ⁻⁷	[26,27,28,29,30]
(NG)	900	172	6.0 x 10 ⁻⁶	
	900	250	6.0 x 10 ⁻⁵	
	900	300	1.0 x 10 ⁻⁴	
Ti-48A1-2Cr-2Nb	760	320	3.0 x 10 ⁻⁶	This study
(DP)	760	215	1.4 x 10 ⁻⁷	Exp 4
	760	245	4.3 x 10 ⁻⁷	
	760	170	2.3 x 10 ⁻⁷	
	760	125	1.6 x 10 ⁻⁸	
	760	145	1.8 x 10 ⁻⁷	
	760	154	3.0 x 10 ⁻⁷	
	760	105	1.8 x 10 ⁻⁸	Exp 7
	760	145	2.0 x 10 ⁻⁸	
	760	155	2.4 x 10 ⁻⁸	
	760	147	2.2 x 10 ⁻⁸	
	760	177	4.1 x 10 ⁻⁸	
	760	155	2.8 x 10 ⁻⁸	
	760	205	5.1 x 10 ⁻⁸	

CHAPTER FIVE

CREEP EXPERIMENTS ON Ti-48AI-2Cr-2Nb

5.1. INTRODUCTION

A systematic study of the creep deformation behavior of investment cast duplex near γ -TiAl, having the chemical composition of Ti-48Al-2Cr-2Nb (at%), has been completed. Because two-phase TiAl alloys have shown to be a promising candidates for high-temperature light-weight structural applications, engineers are interested in the hightemperature creep characteristics. However, there has been only a small amount of research published on the creep properties of TiAl. The values for the different creep parameters for each specific condition was collected and analyzed. This study encompasses all three stages of creep (primary, secondary and tertiary), but an emphasis on the steady-state (secondary) condition is explored to determine the possible creep mechanisms by the values obtained.

In this chapter the chemical composition and heat treatment history is given. A summary of both experimental procedures in air and rough vacuum atmospheres is given. Finally, the results of each experiment is shown as a series of six plots displaying a combination of strain, time, stress, load and strain rate.

5.2. MATERIALS

The creep specimens were investment cast near-gamma Ti-48Al-2Cr-2Nb made by Vacuum Arc Remelting (VAC) and cast at Howmet Corp., Whitehall, MI. The casting technique was accomplished when the ingot was melted in a water-cooled copper crucible and then poured into a preheated ceramic molds. The molds had specially developed ceramic facecoats to minimize the reaction between the ceramic molds and the solidifying γ alloy. The cast rods, 152 mm in length and 16 mm in diameter, where removed from the shell and hot isostatically pressed (HIP'ed) at 1260 °C/172 MPa / 4h (2300°F/25 ksi/4h) to eliminate solidification porosity. The composition of this material is shown in Table 5.1. The material was then heat treated in inert Ar atmosphere at 1573 K for 20 hours and gas fan cooled with a cooling rate of 65 K/min to produce a duplex microstructure with a (gamma + alpha2) lamellar grains plus equiaxed gamma grains. The rods were machined into 62 mm long tensile specimens having a 25 mm gage length and diameter of 5 mm.

Table 5.1 Chemical composition of specimen [33]

Ti	Al	Nb	Cr	Fe	Cu	Si	0	N	Н	
(wt%) bal	32.85	4.47	2.95	0.03	<0.01	0.02	555 ppm	53ppm	23ppm	
(at%) bal	47.4	1.9	2.2							

5.3. EXPERIMENTAL PROCEDURES FOR CREEP EXPERIMENTS

The following is a brief summary of the procedures for creep experiments of TiAl. Since the start of the project our team has completed nine different creep test under different conditions of stress at 760 - 765°C, ranging in time from 31 days to 4 hours. Because the creep experiments were performed either in open-air or in rough vacuum, two different experimental procedures were needed. The experimental procedures and the basic equipment are described in detail in Appendix I

5.3.1. Open-air procedure

Open-air creep testing was performed using an ATS stress relaxation-stress creep frame. Specimen elongation was monitored as a function of time by an linear capacitance displacement transducer (LCDT) attached to the top of the pull rod assembly. To maximize the amount of creep rate information obtained during the study, several experiments employed a stress increment test approach as described previously in the paper. Initial specimen loadings were increased or decreased with a stress interval after allowing enough time for a minimum steady-state creep deformation to occur. The experiment progressed until the creep regime of interest was reached. Then, the specimen was furnace quenched to room temperature while maintaining the final stress and removed for TEM investigation.

5.3.2. Vacuum procedure

Creep testing performed in a rough vacuum also used the same creep frame filled with a 3 inch diameter retort. The specimen elongation was also monitored as a function of time with an LCDT, but the LCDT was placed inside the vacuum chamber and a liquid vacuum feedthrough-cooling system was designed and constructed to keep the LCDT at a constant temperature. Then the LCDT was attached to the specimen by a molybdenum (TZM) extensometer. The temperature of the top and bottom of the specimen was monitored by using a thermocouple vacuum feedthrough. The vacuum condition was approximately 10 millitorr. After creeping the specimen was furnace cooled and removed for TEM investigation.

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5.3.3. Testing techniques

There were several different types of creep experiments that where accomplished. The two most common types of creep testing techniques that where used was the constant load and constant stress creep tests (experiments 1,2,5,6,8,9). However, there where also several experiments that are considered uncommon. One such test was a stress decrement experiment, (experiment 3). Another type of uncommon test employed a combination of stress cycling and creep. This is accomplished by cycling the load up and down as the experiment is creeping (experiments 4,7).

The most common type of test is the constant *stress* creep test. During the test load and strain are used as inputs into the creep frame computer control unit. The control unit then computes the stress from the relation:

$$\sigma = P(1 + \varepsilon)/A_0 \tag{15}$$

where σ = stress, P = load, ε = strain, and A₀ = original x-sectional area.

This equation is used by a microprocessor to adjust the load to maintain a constant true stress. The constant stress creep test causes the load to decrease slightly with increasing strain, due to the above equation.

The constant *load* creep test is different from the constant stress creep test. The constant load creep test results in the stress increasing with increasing strain due to the same relation in equation 15.

As mentioned previously, the assumption that near-gamma TiAl is deformation independent led to the employment of the stress increment technique. This technique allows the investigator to maximize results while minimizing cost. Again, this is accomplished by starting the specimen at a low stress and waiting for a minimum steady-state creep rate to be reached. Then the stress is increased by an appropriate increment and the process is repeated. The uncommon test technique of cycling the load up and down to obtain a combination of a fatigue and creep test uses the same approach as the stress increment technique. However, the two techniques differ when the minimum creep rate is reached the stress is either increased or decreased, unlike the normal convention of a strictly increasing the stress.

5.4 EXPERIMENTAL RESULTS

The most common creep testing technique is to determine the time until failure, and modifications of that particular type of creep testing. The approach this study used was to creep test the material at different stages of creep and at different stress conditions. Then, when the creep regime of interest is reached, to freeze in the mircostructure by furnace quenching. Later the creeped specimen was examined in the TEM [15,33,34] to determine what deformation mechanisms might be operating due to the microstructural evidence observed. There where nine creep experiments on Ti-48Al-2Cr-2Nb completed during this study. Each creep experiment had different test conditions. A detailed account for each and an analysis of the creep data is given in Table 5.2.

Table 5.2
Creep experiments on Ti-48Al-2Nb-2Cr at MSU

Experiment name	Brief description of creep conditions
1. Constant stress:	Load = 760 lbs, stage-two creep performed in open-air. Temp = 765°C, $\sigma = 160$ MPa s, $= 0.05$
	$\text{remp} = 703 \text{ C}, \text{ O} = 100 \text{ MPa}, \epsilon_{\text{f}} = 0.03$
2. Constant load:	Stage-two creep performed in open-air.
	Temp = 765°C, σ = 183 MPa, $\varepsilon_{\rm f}$ = 0.10
3. Load decrease/inc.:	Reload of the specimen in experiment #2
	Load = 1400 lbs, stage-two creep performed in open-air.
	Temp = 760°C, σ = 320 - 150 MPa, $\varepsilon_{\rm f}$ = 0.11
4. Load increase/dec.: **	Stage-three creep performed in vacuum.
	Temp = 760°C, σ = 320 - 125 MPa, ε_{f} = 0.15
5. Constant stress:	Stress = 185 MPa, stage-one creep performed in open-air.
	Temp = 760°C, $\varepsilon_{\rm f} = 0.02$
6. Constant stress:	Stress = 185 MPa, stage-two creep performed in open-air.
	Temp = 760°C, $\varepsilon_{\rm f} = 0.23$
7. Cyclic stress:†	Stage-three creep, performed in open-air.
	Temp = 765°C, σ = 105 - 205 MPa, ε_{f} = 0.17
8. Stress reduction:	Stage-two creep performed in open-air.
	Temp = 760°C, σ =180 MPa, ε_{f} = 0.16
9. Constant load:*	7 vol% TiB ₂ , Load = 801 lbs,
	Stage-two creep performed in vacuum.
	Temp = 760°C, $\varepsilon_{\rm f} = 0.08$
where * = vacua	um experimental procedure
$\dagger = fatiguestical for a final sector for a final $	train
c _f – illai s	

The creep properties of the present work are shown. In Figures 5.1 - 5.9 the plots display the data as: (a) stress vs. strain, (b) strain-rate vs. strain and (c) strain vs. time are shown on the first page of the figure. On the second page, multiple axis graphs show the (d) strain-load vs. time, (e) logarithmic strain-rate vs. logarithmic stress and (f)strain rate-stress vs. strain.

In order to illustrate any trends in the data, Figure 5.10 shows a comparison of all the experiments on one plot of logarithmic strain-rate vs. logarithmic stress. Figure 5.11 shows a comparison of the temperature compensated comparison plot of logarithmic strain-rate vs. logarithmic stress. The temperature compensation technique will be described in detail in the next chapter.



































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Figure 5.10 Comparison of all MSU experiments



Figure 5.11 Comparison of all MSU experiments temperature compensated

CHAPTER SIX

ANALYSIS

6.1. INTRODUCTION

The majority of the literature on creep of TiAl [8,19,20,23,25 - 30,35] is directed at describing the creep behavior as utilizing a single deformation mechanism, based on the concepts of common creep theory. However, because there are so many different microstructures and each has it's own unique properties, a single model for all gamma-based titanium aluminides is unrealistic. Therefore, instead of directing this study toward a single deformation mechanism to explain the creep behavior of TiAl, this study will examine the different creep parameters that surround each creep experiment in an attempt to analyze the trends that appear in the data.

This chapter will consider the unstable microstructure of TiAl and explain why a composite model is useful for understanding the deformation characteristics of TiAl. Then an analysis of the interface between the $L1_0$ and the DO_{19} will provide important implications on the criteria for choosing elements of a composite model. The composite creep model presented by Soboyejo [8] will also be re-evaluated in the light of the interface, and a simplified model based on the application of the Mukerjee-Bird-Dorn equation to the rule-of-mixtures will be presented.

6.2. THE VARIABILITY OF UNSTABLE MICROSTRUCTURE IN TIAI

As mentioned previously, the creep behavior of TiAl is not understood. The creep rate values for specimens of nearly the same composition and microstructure, at the same creep conditions are often different. Furthermore, the values for the activation energy, Q, and the stress exponent, n, reported in the literature survey vary widely from study to another. This leads to confusion about which creep deformation mechanism(s) is responsible for the observed creep behavior of TiAl.

The literature survey has shown that slight changes in Al content of TiAl has a very large effect on the creep behavior. However, slight changes in the heat treatment history, grain size, test temperature, test environment and prior deformation which the creep specimen has experienced also have a large effect on the creep behavior of TiAl. Therefore, the creep data reported in the literature is often incomplete and inconclusive. This illustrates the fact that TiAl has a microstructure that can change very easily with slight changes in composition and/or test conditions.

Furthermore, alloying elements added to TiAl increase the variability of the TiAl microstructure. This is due to the fact that the phases present do not correspond to the phase diagram proposed by McCullough [18](SeeFigure3.3). Because McCullough only considers binary TiAl in the phase diagram, once alloying elements are added to TiAl the phase diagram is no longer valid, and new ternary phase diagrams are needed. For example adding Cr to TiAl causes the alpha-transus line to be depressed, thereby raising the Al content of primary alpha phase. This leads to a reduction in volume fraction and width in the alpha2 lamellae. How much of a reduction with a given concentration of Cr is also variable [8, 23, 25,54]. The creep behavior of these materials is not similar, and the

creep deformation mechanism(s) responsible are not consistent with common creep theories. More work is necessary to understand the effect of alloying on the microstructure of TiAl.

Throughout the literature, the constituents of the microstructure are often generalized without considering the variability of the microstructure. This is why a composite model is important tool for describing the creep behavior of TiAl. The composite model allows for modifications of the description of the creep behavior to account for the variability of the microstructure. While the previous accounts attempt to describe the creep behavior by applying the existing creep data to common creep theory formulas. This approach resulted in a limited success (many of the successful attempts are accomplished by adding fitting parameters, such as modifying the geometrical constant, A). The advantage of employing the composite approach to describe the creep behavior, is the ability to identify different heat treatment histories, grain sizes and test conditions within the constituents of the microstructure. Only the composite model can make a distinction between which constituent(s) is adding to, or subtracting from the instability of the microstructure.

6.3. THE L10 AND DO19 TRANSITIONAL INTERFACE

The lamellar part of the duplex structure has become an important topic of discussion when trying to understand the creep properties of TiAl. The values found in the literature [8,23,25 - 30] for the diffusion rate through the lamellar structure are varied and contradicting. Therefore, it is important to investigate the interface between the $L1_0$ and the DO₁₉ lamellar constituents.

In the literature [8,22,23,25,31] the diffusion rate through the interface is

considered to be controlled by the diffusion along the interface by the two diffusion rates for both the $L1_0$ and the DO_{19} . However, consider the interface between the lamellae constituents consisting of a gradual change from the $L1_0$ (gamma) stacking sequence of ABCABC, to the DO_{19} (alpha2) stacking sequence of ABABAB. Then the interface between gamma and alpha2 can not be considered to have an normal interface boundary. Therefore, the interface between the two lamellae constituents can be thought of as having a *transitional interface*.

Because the chemical composition of the duplex structure is usually in the 45-50 at% Al region, the titanium rich side of the phase diagram, Figure 3.1, the chemical composition of each individual phase is often off-stoichiometric. Based on the phase diagram, the concentration of aluminum atoms to titanium atoms in Ti_3Al is not always two to one. The chemical composition of the different constituents in the lamellar interface evolves in increments from one composition to another, and has a one-half $L1_0$ one-half DO_{19} composition at the so called "interface", thus the idea of a transitional interface. To further explain, it is well known that the stacking sequence of the gamma corresponds to the (111) plane of the $L1_0$ (Figure 6.1) and the stacking sequence of the alpha2 corresponds to the (0001) basel plane of the DO_{19} (Figure 6.2). Then the interface between the gamma and alpha2 is depicted in Figure 6.3.



Figure 6.3. Interface between the gamma and alpha2

But allow the composition of the interface to be off-stoichiometric. Then a titanium atom occupies a aluminum atom site. Note, that the relative size of aluminum and titanium are nearly equal, so therefore very little lattice strain is involved. The transitional interface plane appears as below (Figure 6.4 and Figure 6.5).



Figure 6.4 Transitional interface of TiAl and Ti₃Al (a)



Figure 6.5 Transitional interface of TiAl and Ti_3Al (b)

Thus, the stacking of this transitional interface corresponds to the (111) plane of the $L1_0$ while in the DO_{19} basel plane. This is considered a *slightly disordered matrix*. Where the amount of disorder is controlled by the location away from the transitional interface, which shares a nearly 50%-50% composition. Therefore, the diffusion rate through the interface is not two diffusion rates, for the $L1_0$ and the DO_{19} , controlled by the diffusion along the interface of the two (Figure 6.6). Instead there is a single diffusion rate through the lamellar due to the solid interface nominalizing the diffusion path through the lamellar structure (Figure 6.7). A description of the diffusion rate and Ti content vs. lamellar position is shown in top graph of Figure 6.8.



Figure 6.6 Representation of diffusion without transitional interface



TI-75AI -

Figure 6.7 Effect of Ti concentration on Diffusion



Figure 6.8 Ti content vs. lamellar position

6.4. ANALYSIS OF SOBOYEJO COMPOSITE CREEP MODEL

As mentioned earlier, Soboyejo [8] and others [14,42] report that gamma-

based TiAl alloys with duplex (gamma-alpha2) microstructure can be modeled by recognizing that the two phase alloys are composites that consists of gamma and alpha2 constituents. Because creep deformation within two-phase γ alloys may occur by any one of the

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deformation mechanisms discussed previously, Soboyejo developed a method of determining the creep rate by applying a simple rule-of-mixtures approach. Again, the equation Soboyejo used for the composite strain rate, $(d\epsilon/dt)_c$, is given by [8]:

$$(d\varepsilon/dt)_{c} = f_{\alpha 2}[(d\varepsilon/dt)_{DC\alpha 2} + (d\varepsilon/dt)_{DG\alpha 2} + (d\varepsilon/dt)_{C\alpha 2} + (d\varepsilon/dt)_{NH\alpha 2}] + f_{\gamma}[(d\varepsilon/dt)_{DC\gamma} + (d\varepsilon/dt)_{DG\gamma} + (d\varepsilon/dt)_{C\gamma} + (d\varepsilon/dt)_{NH\gamma}] + (15)$$

6.4.1. Application of the Soboyejo model

When executing the equation for the creep rate, Soboyejo would apply the theoretical values to each the individual creep deformation mechanisms (DC,DG,C,N-H) for both constituents (gamma, alpha2). Then he would compare the value obtained for each deformation mechanisms to the observed creep rate, and the deformation mechanism that was the closest to the observed value was named the dominate creep deformation mechanism. While the other deformation mechanisms that resulted in values that did not come close to the observed creep rate where labeled insignificant and removed from the above equation.

The value for the observed creep rate was $6.44 \times 10^{-7} \text{ s}^{-1}$ at creep conditions of 800° C and 200 MPa. Application of the diffusion-controlled mechanisms showed that the value for Nabarro-Herring type creep was too large when compared to the actual creep rate. Obtaining a value of $120\alpha_{\text{NH}}$ (s⁻¹) for the temperature and stress conditions reported. Coble type creep was one order of magnitude faster than the observed creep rate. When the dislocation controlled mechanisms were applied, the values for the creep rate were much too slow compared to the observed creep rate. However, there was a severe lack of adequate information on the dislocation structures to make a justifiable approximation. Therefore, Soboyejo indicates that Coble creep is the dominate deformation mechanism for the creep conditions reported.

6.4.2. Problems with the Soboyejo model

There where several problems with the actual article published by Soboyejo [8], including leaving out certain characters out of common creep theory formulas (i.e. p, d and T in the Coble creep equation). However, this paper will only address certain theoretical values used in the equations that formulate equation 15, and some basic assumptions that were made by Soboyejo.

Soboyejo listed values for the atomic volume, Ω , and thickness of the grain boundary, δ , listed on Table 6.1. An attempt to reproduce the values indicated in Table resulted in another value for the atomic volume of the DO₁₉ structure. This would have a definite impact on the creep rate calculated by Soboyejo.

Table 6.1Different creep parameter values used by Soboyejo [44]

structure	<u>a</u>	<u>C</u>	Ω	δ
Llo	0.398 nm	0.408 nm	$4.62 \times 10^{-30} \text{ m}^3$	0.001 µm
DO ₁₉	0.462 nm	0.578 nm	2.67 x 10 ⁻²⁸ m ³	0.001 µm
	where $a_s = 1 \ \mu m$ for a typical lath, and $b = 0.398 \ nm$			

note: the DO₁₉ structure has a hexagonal based atomic volume, also there are errors in atomic volume

Also, here seams to be some confusion about the value for the grain size being determined by either the average individual lath size of 15μ m, or the average size of the lamellar colony which is approximately 200 μ m. The correct value for the gram size would be estimated as the size of the colony approximately 200 μ m. Applying the wrong value in the dislocation controlled creep equation would also have a very large effect on the calculated creep rate by Soboyejo.

Soboyejo does indicate that the differences between the predicted and the measured creep rate may be due to the use of Coble creep geometrical constant for materials with duplex microstructures. This leads to an investigation of the assumption that the

equiaxed grains and lamellar colonies are arranged in series as in Figure 4.8. This assumption neglects the highly anisotropic nature of the grain boundary orientation, See Figure 6.9.



Figure 6.9. Idealized highly anisotropic duplex grain

Coble creep utilizes diffusion along grain boundaries. The original assumption indicates that grain boundaries are smooth and easy for a diffusing atom to transverse. However, the new idealized grain shows that the grain boundaries are highly misaligned due to the lamellar grain structure. So, the length of the diffusional path along grain boundaries will be longer and harder for a diffusing atom to transverse. This would cause the diffusion along the grain boundaries to slow down.

Furthermore, another important aspect of the new idealized grain to observe, is that no grain boundary sliding can occur due to the highly misaligned grains. As opposed to the original idealized grain where the grain boundaries where portrayed as smooth and grain boundary sliding easy to initiate.

6.4.3. Three constituent composite creep model

Another way to view duplex TiAl as a composite model is to consider the structure as having three constituents. The three constituents being the equiaxed γ grains, the α_2 phase in the lamellae and the γ phase in the lamellae. The reason for this type of arrangement is due to the fact that α_2 tends to absorb oxygen and other impurities. Because these impurities tend to increase strength and reduce diffusion in the α_2 , both gamma phases can be thought of as being *soft* or diffusion is faster than the *hard* α_2 phase. However, the impurity content is lower in the γ -phase of the lamellar structure than in the equiaxed γ grains because to its close proximity to the α_2 phase. Therefore, the γ -phase in the lamellae would experience the greatest reduction in the impurity content, and would be considered *softer* than the γ in the equiaxed grains. Thus, there is a need for the division of the gamma phase into the gamma in the lamellae and the gamma in the equiaxed grains.

$$D_{c} = F_{\gamma e} D_{\gamma e} + F_{\alpha 2 l} D_{\alpha 2 l} + F_{\gamma l} D_{\gamma l}$$
(16)

where D = Diffusion Coefficient, F = phase volume fraction of γ_e in the equiaxed, α_{21} lamellae and the γ_1 lamellae.

The phase volume fraction would be approximately $F_{\gamma e} = 0.50$, $F_{\alpha 21} = 0.05$ and $F_{\gamma 1} = 0.45$.

However, no information is available on the difference in diffusion rates in the equiaxed gamma phases and the lamellae γ . More work needs to be accomplished in this area.

6.5. RULE-OF-MIXTURE-MUKERJEE-BIRD-DORN EQUATION

As mentioned earlier, Soboyejo [8] and Hazzledine [14] report that the creep behavior of gamma-based alloys with duplex microstructure can be modeled by

recognizing that the two phase alloys are composites that consists of alpha2 and gamma phases. However, instead of using the Soboyejo approach as described previously, an attempt to use more physically meaningful mechanisms and to simplify the mathematics has resulted in a new approach. Consider an approach which combines the composite rule-of-mixtures with the Mukerjee-Bird-Dorn equation. Thus, the new approach yields the equation:

$$(d\epsilon/dt)_{cMBD} = f\alpha_2 \{ (AD_{\sigma\alpha2}Gb/kT)(\sigma/G)^n exp(-Q_{SD}/RT) \} + f\gamma \{ (AD_{\sigma\gamma}Gb/kT)(\sigma/G)^n exp(-Q_{SD}/RT) \}$$
(17)

where f is the phase volume fraction for the α_2 and γ phases. This statement of the MBD equation has no grain size dependence (p = 0).

By utilizing the equations
$$D_{\alpha 2} = D_{\alpha 2} \exp(-Q_{SD}/RT)$$
 and

 $D\gamma = D_{\alpha\gamma} exp(-Q_{SD}/RT)$ for the diffusion rates of α_2 and γ respectively, the above equation becomes:

$$(d\varepsilon/dt)_{cMBD} = f\alpha_2 \{ D_{\alpha 2} (AGb/kT) (\sigma/G)^n \} + f\gamma \{ D_{\gamma} (AGb/kT) (\sigma/G)^n \}$$
(18)

Then using values obtained for the diffusion coefficient reported by Ouchi[31],

$$D\alpha_2 = 5.057 \text{ x } 10^{-8} \text{m}^2 \text{s}^{-1}$$
 and $D\gamma = 1.241 \text{ x } 10^{-6} \text{m}^2 \text{s}^{-1}$

But, recall that diffusion coefficients reported by Ouchi [31] where computed from nonchromium containing alloys. Thus, the values have to be scaled using ratios of phase transformations determined from previous studies. The ratio appears to be by a factor of 10 slower for chromium containing alloys [8,43]. Therefore,

$$D\alpha_2 = 5.057 \text{ x } 10^{-9} \text{m}^2 \text{s}^{-1}$$
 and $D\gamma = 1.241 \text{ x } 10^{-7} \text{m}^2 \text{s}^{-1}$

A study by Schafrik [44] reported a value for the temperature dependent shear modulus. However, the aluminium concentration of the study was 36 (at%) Al in TiAl, and as mentioned previously this has an effect on the properties of the material. Therefore, by an extrapolation, and the fact that we know the shear modulus at room temperature for Ti-48Al-2Cr-2Nb [34] is 69620 MPa, we can estimate a value using the following equation:

$$G = 73494 - 13T = 69620 MPa (T = 298) and 59545 MPa (T = 1073)$$

The Burgers vector, b, is found by considering the different lattice parameters. The gamma (L1₀) structure has a c/a ratio of 1.03 with a = 0.398 nm. Thus, the [110] direction the Burgers vector is SQRT(2)/2 x (0.398 x 10^{-9}) = 0.283 nm. While the alpha2 (DO₁₉) has a hexagonal structure with a = 0.462 nm. However, because the DO₁₉ is considered a super lattice the hexagonal lattice is twice as large as a normal hexagonal closed pack (hcp) lattice. Thus, the burgers vector is b = 0.462 nm. Which implies super dislocation controlled creep.

The geometrical constant A is a fitting parameter based on the data reported by Howmet [25]. A number of geometrical factors are not identified, such as: the grain size and the sub grain size. A is often composed of the relationship between the stacking fault energy and the grain size.

$$A = A'(\gamma/Gb)^3(b/d)^p$$

where γ = stacking fault energy, G = shear modulus, b = Burgers vector, d = grain size, p = grain size parameter.

A list of the values used in the rule-of-mix-MBD equation is shown in Table 6.2.

Table 6.2.	
List of various creep parameter values	

Gamma	<u>Alpha2</u>
Llo	DO ₁₉
$D_{\gamma} = 1.241 \text{ x } 10^{-7} \text{ m}^2 \text{s}^{-1}$	$D_{\alpha 2} = 5.057 \text{ x } 10^{-9} \text{ m}^2 \text{s}^{-1}$
b = 0.689 nm	b = 0.462 nm
G = 59545 MPa	G = 59545 MPa
$\sigma = 400 \text{ MPa}$	$\sigma = 400 \text{ MPa}$
n = 4.5	n = 4.5
$A = 1 \times 10^{-11}$	$A = 1 \times 10^{-11}$
$T = 800^{\circ}C$	$T = 800^{\circ}C$

Therefore, by applying values given we obtain:

For alpha2:
$$(d\epsilon/dt)_{\alpha 2} = D_{\alpha 2}(AGb/kT) * (\sigma/G)^n =$$

(5.057 x 10⁻⁹)(1 x 10⁻¹¹)(59545 x 10⁶)(0.462 x 10⁻⁹)/(1.38 x 10⁻²³)(1073)
* (400 x 10⁶/59545 x 10⁶)^{4.5} = 1.57 x 10⁻⁸ (s⁻¹)

.

For gamma: $(d\epsilon/dt)_{\gamma} = D_{\gamma}(AGb/kT) * (\sigma/G)^n =$

 $(1.241 \times 10^{-7})(1 \times 10^{-11})(59545 \times 10^{6})(0.689 \times 10^{-9})/(1.38 \times 10^{-23})(1073)$

* $(400 \times 10^6/59545 \times 10^6)^{4.5} = 5.74 \times 10^{-7} (s^{-1})$

Now employing the rule-of-mixture for the nominal volume fraction of 5% α_2 present in the duplex microstructure yields:

$$(d\epsilon/dt)_{cMBD} = (0.05)(1.57 \text{ x } 10^{-8}) + (0.95)(5.74 \text{ x } 10^{-7}) = 5.53 \text{ x } 10^{-7} \text{ (s}^{-1})$$

When compared to the observed value of $6.0 \times 10^{-8} (s^{-1})$ at 800° C and 200 MPa [8], we find the experimental error is

{
$$(6.0 \times 10^{-8} - 5.5 \times 10^{-7})/6.0 \times 10^{-8}$$
} x 100 = 816.7% error.

This is within an order of magnitude of the observed steady-state creep rate, and is approximately the value Soboyejo obtained using the composite creep approach, with Coble creep being the dominate creep mechanism.

However, when compared to the Wheeler [25] observed rate 6.0×10^{-7} at 800° C and 200 MPa, we find the experimental error is

{
$$(6.0 \times 10^{-7} - 5.5 \times 10^{-7})/6.0 \times 10^{-7}$$
} x 100 = 8.3% error

Most mathematical creep models that fall within one order of magnitude of the measured creep rate are considered a decent approximation. The A value was chosen to get a good approximation. The differences between the measured and predicted creep rates in the rule-of-mixture-Mukerjee-Bird-Dorn equation may be due to significant errors in the diffusion coefficients used in the calculations.

When comparing this new approach to the Soboyejo model it is important to mention that Soboyejo only examines a limited creep regime of one temperature and one stress for his creep model. Therefore, it represents a single point when plotted as log steady-state strain-rate vs. log stress. So, in order to compare the new approach more accurately, a range of the predicted creep behavior using the composite-MBD equation is provided. The predicted creep behavior using the composite-MBD equation is shown in Table 6.3.

Table 6.3.					
List of predicted cree	behavior using compos	ite-MBD equation			

<u>Temp.</u>	σ	G	$\underline{D}_{\alpha 2}$	<u>D</u> γ	<u>(dɛ/dt)</u>
(°C)	(MPa)	(MPa)	(m^2s^{-1})	(m^2s^{-1})	(s ⁻¹)
700	100	60845	1.59 x 10 ⁻¹⁰	3.91 x 10 ⁻⁹	3.0 x 10 ⁻¹¹
700	200	60845	1.59 x 10 ⁻¹⁰	3.91 x 10 ⁻⁹	3.0 x 10 ⁻¹⁰
700	300	60845	1.59 x 10 ⁻¹⁰	3.91 x 10 ⁻⁹	2.0 x 10 ⁻⁹
700	400	60845	1.59 x 10 ⁻¹⁰	3.91 x 10 ⁻⁹	2.0 x 10 ⁻⁸
800	100	59545	5.057 x 10 ⁻⁹	1.241 x 10 ⁻⁷	1.0 x 10 ⁻⁹
800	200	59545	5.057 x 10 ⁻⁹	1.241 x 10 ⁻⁷	3.0 x 10 ⁻⁸
800	300	59545	5.057 x 10 ⁻⁹	1.241 x 10 ⁻⁷	7.0 x 10 ⁻⁸
800	400	59545	5.057 x 10 ⁻⁹	1.241 x 10 ⁻⁷	6.0 x 10 ⁻⁷
900	100	58245	8.90 x 10 ⁻⁸	2.19 x 10 ⁻⁶	2.0 x 10 ⁻⁸
900	200	58245	8.90 x 10 ⁻⁸	2.19 x 10 ⁻⁶	3.0 x 10 ⁻⁷
900	300	58245	8.90 x 10 ⁻⁸	2.19 x 10 ⁻⁶	1.0 x 10 ⁻⁶
900	400	58245	8.90 x 10 ⁻⁸	2.19 x 10 ⁻⁶	1.0 x 10 ⁻⁵

Figure 6.10 shows a comparison of different creep conditions on one plot of logarithmic strain-rate vs. logarithmic stress. While Figure 6.11 shows a comparison of the temperature compensated comparison plot of logarithmic strain-rate vs. logarithmic stress. The temperature compensation technique will be described in detail later in chapter 7.









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CHAPTER SEVEN

DISCUSSION

7.1. INTRODUCTION

In this chapter, the data from the experiments described in chapter 5 are compared to other studies in the literature [8,25 - 30]. An important issue resulting from this comparison is the assumption of the deformation history independence in multiple stress jump creep tests. The experimental data is normalized by the temperature dependence of the self diffusivity [32] and the shear modulus [44] for most of the available studies on creep of TiAl [8,19,23,25 - 30,35,40,49,60,62]. The data from several studies [19,25 - 30] is compared in plots of logarithmic strain rate vs. logarithmic stress in order to investigate the stress exponent, n. Finally, the mechanisms that soften the microstructure and restricts dislocation glide by lamellae are explored to improve our understanding of the deformation mechanism(s) responsible for the creep behavior of TiAl.

7.2. COMPARISON OF MSU DATA TO LITERATURE

The comparison of the data from this study and the literature has shown that some creep specimens appear to creep faster than others, even-though the creep specimens had the same nominal composition and the same test conditions. The reasons behind this unusual creep behavior will be explored.

Table 7.1 lists the minimum steady-state creep rates obtained for a given stress and temperature condition for all the creep experiments of this study. The strain at

which the creep specimens achieved minimum steady-state is also given. This information is a useful tool to understand the creep condition of the specimen during deformation.

From the data given in Table 7.1 a trend of increasing steady-state strain rate with increasing stress holds for all the experiments. However, there appears to be two groups of creep behavior. The first group of experiments (1, 2, 3, 4, 8, 9) have a steady-state creep rate that is a magnitude faster than the second group (6, 7).

The steady-state creep rates of group one are approximately equal to the values obtained by Soboyejo [8] $6.4 \times 10^{-7} \text{ s}^{-1}$ for creep conditions of 800°C and 200 MPa and the values obtained for group two are approximately equal to the values obtained reported by Wheeler [25] $6.0 \times 10^{-8} \text{ s}^{-1}$ for the creep conditions of 815°C and 200 MPa. It is curious that both trends in this study have similar data existing in the literature. However, due to the fact the other creep studies [8,25] had specimens of the same composition and similar microstructure, and were tested at similar creep conditions, the data seems contradictory.

<u>Composition (at%)</u>	<u>#</u>	<u>T (°C)</u>	<u>σ (MPa)</u>	(dɛ/dt) _{min}	<u> </u>
Ti-48Al-2Cr-2Nb		760	160	1.0 x 10 ⁻⁷	0.05 _f exp 1
(DP)		760	183	6.0 x 10 ⁻⁷	0.10 _f exp 2
		760	315	3.0 x 10 ⁻⁶	0.022 exp 3
		760	225	1.0 x 10 ⁻⁶	0.05
		760	180	6.0 x 10 ⁻⁷	0.076
		760	150	3.0 x 10 ⁻⁷	0.095
		760	175	3.4 x 10 ⁻⁷	$0.107_{\rm f} (0.13_{\rm f})^*$
	1	760	320	3.0 x 10 ⁻⁶	0.045 exp 4
	2	760	215	1.4 x 10 ⁻⁷	0.065
	3	760	245	4.3 x 10 ⁻⁷	0.073
	4	760	170	2.3 x 10 ⁻⁷	0.108
	5	760	125	1.6 x 10 ⁻⁸	0.115
	6	760	145	1.8 x 10 ⁻⁷	0.133

154

186

186

105

145

155

147

177

155

205

180

180

190

3.0 x 10⁻⁷

5.5 x 10⁻⁸

8.5 x 10⁻⁸

1.8 x 10⁻⁸

2.0 x 10⁻⁸

2.4 x 10⁻⁸

2.2 x 10⁻⁸

4.1 x 10⁻⁸

2.8 x 10⁻⁸

5.1 x 10⁻⁸

2.8 x 10⁻⁷

3.8 x 10⁻⁷

1.0 x 10⁻⁷

0.15_f

 0.23_{f}

0.042

0.053

0.068

0.086

0.105

 0.17_{f}

 0.155_{f}

0.035 exp 8

 $0.08_{\rm f}$ exp 9

0.04 exp 6

0.033 exp 7

Table 7.1 List of creep data from MSU study

where # = data point number

f = final strain

* = reload of exp 2 added to exp 3

7

1

2

3

4

5

6

7

760

760

760

760

760

760

760

760

760

760

760

760

7.2.1. Experiment #4

Experiment #4 employed the stress decrement technique. However, the experiment also cycled the load up and down as the specimen deformed, to obtain a combination of stress cycling and stress decrements during creep testing. The stress was cycled up and down, but a general stress decrease was the overall direction of the experiment (See Figure 5.4 and 7.1). Also, this experiment was performed in a rough vacuum, which has a small effect on the creep behavior (due to reduced oxide formation).

Experiment #4 was of particular interest because it exhibits similar creep properties as found in the studies by Wheeler [25] and by Soboyejo [8] in the same specimen. The trend in common with Wheeler [25] is a distinct change in the stress exponent from n = 1.7 at low stress to n = 7.7 at higher stresses(See Figure 7.1). But there is a large strain between the data points that indicate the n = 1.7 value. The stress exponents here are not characteristic of stress-assisted diffusion creep process, such as Nabarro-Herring or Coble creep, which typically have an exponent of n = 1, or dislocation creep processes which have an exponent of n = 5.

However, the steady-state creep rate in experiment #4 was $2.3 \times 10^{-7} \text{ s}^{-1}$ at conditions of 760°C and 170 MPa which is approximately equal to the value found by Soboyejo [8] of approximately 6.4 x 10^{-7}s^{-1} at a creep conditions of 800°C and 200 MPa(See darken line in table 7.1). But the temperature difference between the two studies is large enough to cause significant differences in the data. Wheeler [25] reports to have a steady-state creep rate of 6.0 x 10^{-8} s^{-1} for creep conditions of 815°C and 200 MPa. Therefore, experiment #4 shares the unique stress exponent change behavior found in the investigation by Wheeler (Wheeler's report was categorized as group two), but had a



Figure 7.1 Unique power-law behavior shared by Experiment #4 and Wheeler [25]

steady-state creep rate of group one.

7.2.2. Experiment #7

Experiment #7 also employed the stress change technique, and the experiment also used an uncommon test technique of cycling the load up and down to obtain a combination of a stress cycling during creep testing. However, instead of the stress direction being generally decreasing overall as in experiment #4, experiment #7 had a generally increasing stress condition(See Figure 7.2). Also, experiment #7 was performed in an open air atmosphere and not in a rough vacuum, as was the case in experiment #4.

The results indicate that the steady-state creep rate obtained is similar to group one, $4.1 \ge 10^{-8} \text{ s}^{-1}$ for creep conditions of 760°C and 177 MPa, which is near the creep rate reported by Wheeler [25], $6.0 \ge 10^{-8} \text{ s}^{-1}$ for the creep condition of 800°C and 200 MPa(See darkened line in table 7.1). However, the experiment did not show the unique power-law behavior shared with experiment #4 and the data from Wheeler [25](See Figures 4.5 and 7.5). A comparison plot of experiments #4 and #7 is provided in Figure 7.3. A temperature compensated plot of the same experiments is provided in Figure 7.4.

7.2.3. Investigation of deformation history independence

Before experiment #4 began, the TiAl specimen has already experienced a complicated heat treatment history. The creep experiment started with loading to a high stress, which introduced dislocations and twinning into the material. This could be considered as another thermomechancial treatment. There can be two types of deformation







Figure 7.3 A comparison plot of experiments #4 and #7



Figure 7.4 A comparison plot of experiments #4 and #7 temperature compensated

mechanisms operating at the same time. One of the mechanisms is the dislocation glideclimb type and the other is a twin softening type. The dislocation glide-climb type of deformation mechanism would exhibit dislocations that are climbing and annihilating each other. But the softening type of deformation mechanisms could result from the mechanical twins that propagated across the grain, and permanently changed the microstructure. Therefore, the material creeps faster, and the assumption of the deformation history independence does not hold.

In the second case, the generally increasing stress experiment (experiment #7) starts off at a low stress and is increased to a high stress, the amount of mechanical twins propagating across the grain is smaller, and the dislocations that are climbing and annihilating remains the same. Therefore, the material creeps slower than the generally decreasing stress group, and the assumption of the deformation history independence holds.

In conclusion, the assumption of the deformation history independence depends on the overall stress direction of the creep experiment. Furthermore, the effect of twinning deformation mechanisms prevents the assumption from being accurate in the generally increasing and decreasing stress directions.

7.3. COMPARISON OF CREEP DATA PLOTS of TIAI

The experimental data has been normalized by the temperature dependence of the diffusively and the shear modulus for all the various creep experiments at different temperatures. The temperature normalization of values allows the investigator a more accurate comparative view of the stress dependence on the creep deformation.

7.3.1. Unique power-law behavior of Ti-48Al-2Cr-2Nb

As mentioned previously, the steady state creep rates plotted in Figures 7.5 and 7.6 reported by Wheeler [25] for Ti-48Al-2Cr-2Nb, reveal a unique power-law behavior within the stress regime tested. A distinct change in stress exponent from n = 3 at a low stress to n = 7 at higher stresses is observed, indicating a change in the controlling creep deformation mechanism. Again, the stress exponents measured here are not characteristic of stress-assisted diffusional creep processes, such as Nabarro-Herring or Coble creep where n = 1, or dislocation creep processes, where n = 5.

Recent work has shown that stress exponents can be increased by a factor of two when pipe diffusion is rate-controlling during intermediate temperature creep deformation [25,43]. With this knowledge, the power-law behavior identified here could be explained by diffusional creep at low stresses with a change to dislocation creep at high stresses, both of which being controlled by diffusion via dislocation pipes. This hypothesis is supported by the observation of dislocation alignment and subgrain formation during the creep deformation of gamma alloys [25]. Also, the existence of misfit dislocations in the alpha2-gamma interfaces can provide regularly spaced fast diffusion paths.








Significant dynamic recrytallization has been observed in the gage sections of specimens creep-tested to failure at 815°C. It is interesting to note the tendency for the recrystallized grain to nucleate along grain boundaries of the initial microstructure. This tendency indicates that grain boundary sliding also could be playing a role in the deformation process at larger strains, since the degree of recrystallization is known to be a function of local plastic strain [25]. Similar examination of specimens deformed at 760°C revealed very little, if any, evidence of dynamic recystallization, even at high stress levels. However, since recrystallization is a function of both temperature and stored strain energy, the critical temperature for dynamic recrystallization at lower plastic strains is likely to be somewhat higher.

An interesting comparison of Ti-48Al-2Cr-2Nb is shown in Figures 7.7 and 7.8 by Hayes [36]. The figures indicates that at 700°C the stress exponent has a value of n = 7. However, at 800°C the stress exponent is between n = 3 and n = 7, and at 900°C the exponent has a value of n = 7. Therefore, the change in the stress exponent is a function of temperature as well as the strain, strain rate and applied stress.









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6.7.2. Oikawa and multiple creep deformation regimes

When comparing the Ti-48Al-2Cr-2Nb (DP) to the composition of Ti-53.4Al (NG) an interesting trend appears. Figures 7.9 and 7.10 show the steady-state creep rates of Ti-53.4Al reported by Oikawa [26,27,28,29,30]. Another distinct change in the stress exponent occurs. However, the stress exponent value changes from n = 8 at low stresses to n = 4.5 at higher stresses. This is the opposite of the creep behavior of characteristic of stress-assisted diffusional creep processes, where the stress exponent shifts from a low value to a high value. Therefore, the creep deformation mechanisms change and the activation energy is changed from 300 kJ/mol to 600 kJ/mol. Note: the last value given for the activation energy is extremely large compared to others found in the literature (See Appendix II).

One possible answer is that there is more than one stress exponent change possible and the data observed is really only one region of the creep regime. Figure 7.11 reveals that for different compositions and microstructures the stress regime for creep deformation mechanism(s) are not the same. However, creep trends still may be viewed only a part at a time. More than one deformation mechanism may be acting at the same time or in concert with another mechanism.



Multiple deformation mechanism for different stress regimes



Figure 7.9 Steady-state creep rates of Ti-53.4Al reported by Oikawa [26 - 30]





7.3.3 Temperature compensation comparisons

The temperature normalization is accomplished by dividing the data for the strain rate (dɛ/dt) (s⁻¹), by the diffusion rate, $D_T(m^2s^{-1})$, at the temperature reported. Then dividing the stress, σ (MPa), by the shear modulus, G (MPa). The values used for the normalization are based on the study by Kroll and Mehrer on Ti-54Al self-diffusion[32] for the diffusion rate, and the temperature dependent shear modulus is based on a study by Schafrik [44]. To Illustrate that temperature normalization is a useful tool, Figure 7.12 shows the data from a hot tension test of Ti-48Al-2Cr-2Nb (DP) at different temperatures reported by Shih and Scarr [23]. The data organized in this way appears the n value slightly decreases with increasing temperature. However, Figure 7.13 reveals the true stress exponent by removing the temperature dependence. The plot also clearly shows the behavior of power-law breakdown located at the top due to the high strain rates of the tension test.

The first global creep plot is shown in Figure 7.14. It shows a comparison of the observed steady-state data from authors Soboyejo [8], Shih and Scarr[23] and Es Souni [60]and represents Ti-48Al-2Cr-2Nb in different stress conditions. All the data except the values included in Figure 7.13 are assumed to obey power-law creep. Figure 7.15 shows the same plot temperature compensated. Again, the temperature compensation gives us a basis to compare different compositions under different stress and temperature conditions. An inspection of Figure 7.15 shows a distinct change in stress exponent from n = 3.7 at a low stress to n = 7.5 at higher stresses is observed, similar to the data by Wheeler [25], indicating a change in the controlling creep deformation mechanism.











Global plot #1 comparing data of authors Soboyejo[8], Shih and Scarr [23] and El Souni [60] Figure 7.14





The second global creep plot was built from the superposition of several temperature compensated creep plots from the following authors: Global creep plot #1 [8,23,60] Figure 7.15, Oikawa [26 - 30] Figure 7.16, D. Wheeler [25] Figure 7.17, A. Loiseau and A. Lasalmonie [35] Figure 7.18, H. Lipsitt [40] Figure 7.19, S.C. Huang [19] Figure 7.20, P. Martin [49] Figure 7.21, M. Bartholomeusz [62] Figure 7.22 and the data from this study Figure 7.23. The second global creep plot is shown in Figure 7.24. This plot is one of the first to show the observed steady-state creep rates, for different stress and temperature conditions, of most of the available data on creep of TiAl.

These studies indicate that the steady-state creep deformation of investment cast gamma titanium aluminide reveals a unique power-law behavior at intermediate temperature and stress levels. The results suggest that a stress-assisted diffusional creep mechanism occurs at low stresses and a dislocation creep mechanism occurs at high stresses. However, the measured activation energy for creep deformation was greater than that required for interdiffusion within single phase gamma, indicating that creep in these alloys may not be explained by intrinsic diffusional processes alone. Dynamic recrystallization was observed in specimens deformed about 2% strain and at high temperatures [45] could be one possible explanation for the large variances in the values obtained. Another explanation is the twin type of deformation. It has been reported that twinning occurs readily in gamma-based TiAl containing chromium[34]. Also, Jin [15] states the twinning that occurs in "parallel" with the creep mechanism and acts as a cross-slip mechanism. When a dislocation is gliding it encounters an obstacle, a mechanical twin can nucleate in the form of the 1/6[112] partials, will slip on different planes where it may recombine with







Figure 7.17 Wheeler [25] for Ti-48A1-2Cr-2Nb temperature compensated















Figure 2.21 Martin [49] temperature compensated













another partial and continues to slip. Another theory is the 1/6[112] partials nucleate twinning [38]. However, a creep deformation mechanisms that describes twinning does not yet exist. However, the results of this study suggest that the mechanism(s) of creep deformation is cast gamma titanium aluminides is a complex process which depends strongly on the specific stress and temperature regime being examined.

7.3.4. Mechanisms that soften the microstructure or restrict glide

The representation of the second global temperature compensated plot is given in Figure 7.25. The plot shows the solute atom drag type of deformation mechanism as the stress exponent line that is darkened. The data points above the line are believed to be mechanisms that soften the microstructure. The mechanisms that can soften the microstructure include the twinning type of mechanisms that is occurring in parallel with the climb, as discussed previously. Pipe diffusion is another type of mechanism that softens the microstructure. This is due to the fact that the dislocations collect in the in the interface of the alpha2 and gamma phases to take up the misfit parameter. Thus, they provide pipe diffusion to those sections of the alpha two-gamma interface and provides a basis for the double amount of activation energy associated with such a pipe diffusion correlated deformation mechanism.

The data below this line have mechanisms that restrict dislocation glide. The data points on this line have microstructural constraints that restrict dislocation glide. The fully-lamellar type of microstructure has this type of effect on the creep behavior.



CHAPTER EIGHT

CONCLUSION

Titanium aluminide TiAl based materials have become promising candidates for high temperature service. This is due to their high specific strength, stiffness at elevated temperatures, and good oxidation and creep resistance. Also, their density is 2.5 times lower than current nickel-based superalloys. However, very little research of near γ -TiAl based alloy creep behavior has been accomplished, and the creep deformation mechanisms have not been clearly identified. The activation energies are much larger than those for self-diffusion and interdiffusion in TiAl, which implies that the creep rate may be controlled by processes other than the usual lattice diffusion mechanism. The value of the stress exponent varies widely from about 2 to 8 in the literature, which suggests that several deformation mechanisms are involved in the creep of TiAl.

This study has introduced several theoretical arguments, including the combining the composite rule-of-mix and power-law Mukerjee-Bird-Dorn equation as a mathematical creep deformation model and the transitional interface between the gamma and alpha2 lamellar constituents. A literature review has been accomplished and the creep characteristics of different compositions was explored at different test conditions in order to list the trends that appear in the data. Several creep experiments of Ti-48Al-2Cr-2Nb have also been achieved and the data obtained was then compared to the data in the literature. The results of the experiments indicate that the assumption of the material being stain deformation independent can be questioned, depending on the general stress direction. Additionally, As a part of ongoing research concerning creep deformation mechanisms, this study has investigated the collective creep data from other investigations. The data was plotted as logarithmic strain rate vs. logarithmic stress in order to investigate the stress exponent, n. Later, the experimental data was then normalized by the temperature dependent diffusivity [32] and the temperature dependent shear modulus [44] for the various creep experiments at different stresses and temperatures. The temperature normalization of values allows the investigator a more accurate view of the stress dependence on the creep deformation. Finally, a discussion of the mechanisms that soften the microstructure or restrict glide was explored to improve our understanding of the deformation mechanism(s) responsible for the creep behavior of TiAl.

This work was directed at solving some of mystery surrounding the creep behavior of this material. However, the details of deformation mechanisms are not known and the limited results in the literature are not consistent with the creep theory. More research is needed to understand the creep behavior of TiAl.

APPENDIX I

EXPERIMENTAL CREEP PROCEDURE

A.1. INTRODUCTION

This part of the appendix describes the development of facilities and procedures for creep experiments of Titanium aluminides. The assignment was to produce a series of high-temperature creep experiments of titanium aluminides, under different creep conditions and different atmospheres. The project is under the direction Dr. Thomas R. Bieler and is in cooperation with Howmet Corporation International, Whitehall, Michigan, under the supervision of Dean Wheeler.

Since the start of this project we have completed nine different creep test under different conditions, ranging in time from 31 days to 4 hours. The experimental procedures for the creep experiments are discussed and the basic equipment used is listed for reference. It was interesting to notice that, as the creep experiments progressed our team kept on expanding and modernizing the creep equipment used to produce more accurate results. The creep frame design changed dramatically for the last experiment that called for an entirely different set of experimental procedures. So, the experimental procedures will be broken into two sections the old and the new procedures, respectively.

When starting this project some test equipment had already been purchased. However, the equipment had never been used before and it was necessary to design and construct several mechanisms to enable the test equipment to be able to

produce creep results. Some of these projects ranged from building a cooling system for the creep frame to constructing vacuum electric and liquid feed-throughs.

The system used for the creep experiments is an ATS series 2710 creep tester. The system is designed to perform either creep testing (constant load) or stress relaxation testing (constant strain). Furnished with 20 to 1 ratio lever arm to provide high load capacity. A list of the equipment used for the creep experiments is listed below (See Figure 22)

A.2. LIST OF CREEP FRAME EQUIPMENT

Linear capacitance transducers (LCT) - allows a small change in length to be measured as a change in capacitance:

$$dl = h/0.4 ldC$$

where h = dist. btwn cap. plates, l = length

Because the capacitance of the LCT will also change with temperature the LCT will be insulated with a cooling jacket. If the LCT is not insulated it will result in the change in temperature acting as a change in length and produce error. There are two acting LCTs on the creep frame, there are two because if one fails there will be a backup. The LCT model L1-12 was purchased form Applied Test Systems (ATS). The LCT's where manually calibrated to find the sensitivity.

Load cell - allows a change in length to be measured as a change in resistance. The load cell acts as a transducer to measure the resistance and translate the value into a load. The change in resistance of a given object is[61]:

$$dR = \rho(dl/A)$$

where dR = change in resistance, ρ = resistivity, dl = change in length, A = area. The load cell was manually calibrated.

Draw Head and the Jack work together as a team to apply a load on the creep specimen. The draw-head is attached to the lower pull rod system by the load cell. The draw head is most commonly used in situations where rapid loading or unloading is needed such as in initial load up and thermal expansion. The jack is the attached to the upper pull rod system by a long chain that is connected to a 20 to 1 lever arm. The jack is used mostly for very small adjustments in loading or unloading during a creep experiment. Both the jack and the draw head can be controlled either manually or by the machine. Therefore, it is referred to as either machine controlled or manual controlled. During machine controlled the machine uses set points to define the upper limit, middle set point, and lower limit boundaries. The draw head and jack can also be operated manually.

Creep frame control unit - allows the user a choice of different options of creep test experiments. The dials controlling the speed of the jack and the draw head in machine controlled loading are labeled "ramp" and "control". These dials are located on the display rack and control the speed of either the jack or the draw head depending which is in use. The ramp governs the speed of the loading until the set point has been reached. Then the control dial settings take over and regulate the speed of the loading device. After the desired load has been attained the control will ensure that the load will be kept stable with a limited cycling between upper and lower set points. The Creep frame controller was purchased from Applied Test systems INC. (ATS) and Electronic Instrument Research Corp., (EIR) model number SS-100A.

Constant stress control unit - allows the user to switch from strain output to stress (psi) output. The measurements from the LCT and the load cell are inputted into the control unit and a display panel reads out the strain, stress and/or load. The constant stress control panel allows the user to be able to produce many creep experiments including: constant load creep, constant stress creep, constant strain rate creep as well as load reduction/ increase creep experiments. For example for an constant stress creep experiment, the control unit has an input for the initial length and cross sectional area, and then uses the change in length to compute the true cross-sectional area to maintain a constant stress on the specimen.

Extensometer - allows the LCT to be directly attached to the creep specimens during the creep test. Thus, the value for the change in length is more accurate and more sensitive. The extensometer is made from a molybdenum based material called TZM and has excellent properties at high temperatures in an vacuum or inert atmosphere. However, in open-air these properties are degraded by oxidation and forms MoO₃ [56].

Pull rods - there are two pull rod systems on the creep frame, the upper and lower pull rods systems with respect to the position of the specimen. Both are made from stainless steel with 3/4" -10-2A Threaded ends. Both systems have one hollowed out pull rod that attaches to the cooling system. This provides cooling to the inner most pull rods. The pull rods are equipped with heat baffles to control thermal conduction during the vacuum creep experiment.

Grippers- connects the creep specimen to the pull rod systems. The grippers are made from a Ni-based super alloy. This provides the creep frame with a system that can insulate the creep specimens from the cooling from the pull rod system.

Furnace - allows the specimen to reach temperatures as high as 1200°C. The furnace is electrically power and is controlled by a furnace control unit. The furnace used was unique because it has three zones. That is, the furnace possesses three small furnaces within one large furnace, the three smaller furnaces are located top, middle and bottom of the large furnace. This gives the user the ability to adjust the furnace temperature relative to the position of the specimen in the furnace. However, there is only one thermocouple port located in the middle of the heat zone to monitor all three zones.

Furnace control unit - allows the user to operate or program the furnace. The furnace control used was a mircoprocessor based general purpose control unit. This instrument has the start/stop, auto/manual, ramp and soak and multisetpoint functions. The furnace has a set point control that reacts to the thermocouple input from the furnace. The furnace control unit was purchased from LFE and is model 2010.

Thermocouple readout panel - allows the user to monitor the temperature of the top and bottom of the specimen, as well as the transducers. By attaching the thermocouple wires to the panel a dial is turned to monitor one thermocouple at a time.

Data collection system - The first data collection system consisted of an oscilloscope but later was upgraded to a computer data collection system.

(a) The oscilloscope was used to monitor the load and strain vs. time continuously. The oscilloscope has a memory system that enables the user to record the data on a floppy disk. From the information given on the readout, a strain-rate can be calculated by hand. The oscilloscope is made by Nicolet Corp. and is model 4095 B.

(b) The computer data system is used only in the very last experiment. The computer was not only able to record the strain and load vs. time, but could also

record the temperature and the strain-rate of the specimen. The computer has the ability to analyze the data and produce a output so that strain-rate calculations done by hand is not necessary. The computer used was an IBM Personal System/2 Model 30 286 with an Intel model 287 XL coprocessor chip installed to maximize the computer's efficiency.

A.3. OPEN-AIR CREEP EXPERIMENTAL PROCEDURE

The open-air creep experimental procedure is as follows:

1. Apply antisieze compound. Before assembling the upper and lower pull rod systems, the threading of the different pull rods, fittings, grippers and specimen have to be smeared with a molybdenum disulfide based grease. This is to prevent the pull rods and the fittings, grippers and the specimen from bonding together as a result of the high temperature.

2. Assemble the pull-rod system. Connect the upper pull rod system with the upper gripper and attach the cooling connection. Place the extensometer bar at the top of the assembly, and screw entire pull rod system into the lever arm connection. Assemble the lower pull rod system and put on the cooling connection. Attach the specimen to the lower pull rod gripper. Then, carefully screw the specimen into the upper gripper and connect the bottom of the lower pull rod system into the top attachment of the draw head.

3. Transducer assembly. After the pull rods are in place. attach the transducers to the top of the creep frame underneath the extensometer bar. The transducer pins need to be compressed against the bar. 4. Connect the transducer cooling jackets. By moving the copper cooling jacket around the transducer and then connecting to the main cooling systems, the transducers will be kept at a constant temperature.

5. Attach the cooling connection to the pull rods. There are two holes on each side of the hollow pull rods. Line up the top and bottom tubes of the cooling connection with the holes in the pull rod.

6. Turn on load control. To keep the specimen from experiencing compression it is a good practice technique to turn on the load control and keep the system at a low load. The procedure for load control is:

a) set machine control to standby

b) set the three set points (high, middle, low)

c) set machine control to run

7. Attach thermocouple wire to the specimen. Connect two high temperature thermocouple wires to the specimen, place one on the top of the specimen and one on the bottom. Then connect the end of the wire into a thermocouple readout panel. If the connection is bad a "EEE" will appear on the readout.

8. Turn on the furnace. Pull the furnace mounting arm bracket out and center it around the pull rod system. Pack kaowool insulation around the edges of the furnace and the pull rods. Turn on the power to the furnace and furnace controls. There are two temperature readouts, the top right one is the furnace temperature and the bottom middle is the set point. It is important to terrace the furnace temperature up by a gradual increase, to prevent a temperature overshoot that might damage the furnace. 9. Balance the furnace temperature. The top and the bottom specimen temperate reading need to be within one degree of each other. Because heat rises the top of the specimen is usually hotter than the bottom. The three zone furnace helps by regulating the top, middle and bottom furnace temperature. The creep specimen location is also important with respect to the furnace, it should be centered in the middle of the furnace as much possible.

10. Start data collection system. Turn on the oscilloscope or the computerized data collection system. It is important that the output voltage of the transducers be

more positive than -10 volts because that is the limit on the creep control unit output.

11. Start the experiment.

The procedure to start the creep experiment is listed below:

- a) set machine control to standby
- b) place the machine in draw head control
- c) set the three set points (high, middle, low)
- d) set machine control to run
- e) turn on time elapsed recorder on creep frame
- f) record the clock time and time on data collection system.
- g) after the initial load up is finished place the machine control to standby
- h) place the machine in jack control
- i) set machine control to run

A.4. PROCEDURE TO SHUT DOWN OPEN-AIR

When the creep experiment is finished creeping the procedure to experi-

mental shut down procedure is listed below:

1. Stop the experiment.

The procedure to stop the creep experiment is listed below:

- a) set machine control to standby
- b) place the machine in draw head control
- c) place the system on load control
- d) set the three set points (high, middle, low)
- e) set machine control to run
- f) turn off the furnace and move away from pull rods
- g) let the furnace sit overnight
- h) turn off water
- i) turn off data acquisition system
- j) turn off creep control unit
- k) disconnect thermocouple wires
- 1) disassemble pull rod system
- m) remove specimen

A.5. VACUUM EQUIPTMENT

A vacuum system is a technique that attempts to isolate a given space and to evacuate its gas content. The degree of vacuum obtained in a system corresponds to the specific system that is used. The type of vacuum system used for the creep experiments was a positive displacement pump more commonly known as a roughing pump(See Figure 23). As the name implies, the vacuum is only a rough vacuum and not a high vacuum. The main components of the vacuum system are:

Roughing pump. The roughing pump is responsible for creating (pulling) a vacuum inside the vacuum chamber. The pump works by trapping a volume of gas from the chamber to be evacuated, then compresses the volume into a smaller one. Then the discharge of the gas through an exhaust valve. The pump was borrowed from mechanical engineering department, and is a Royal Vac pump model 500.

Foreline. The foreline is the part of the vacuum system that connects the roughing pump to the vacuum chamber. The foreline used was built from thick wall rubber hose. This allowed some flexibility in the system and dampens the vibrations of the vacuum pump.

Foreline trap. The foreline trap is a device that is designed to capture large molecules and remove them from the vacuum system. The foreline trap is used to protect the vacuum system from the backstreaming of roughing pump oil during operation. A foreline trap requires a foreline *bakeout* before each use of the system. The trap is heated up with a electric heater and the captured molecules are then evaporated from the trap.
The vacuum chamber. The vacuum chamber is made up of five constituent chambers that fit together. Each of the vacuum chambers is made from stainless steel. The chambers have o-rings that are made from Viton. The purpose of the o-rings is to seal one chamber to the next. However, for every seal on the chamber there is a possibility for a leak. So by increasing the amount of chambers the possibility for a leak increases. The five chambers are listed below:

Lower and upper bellows. There are two bellows on the vacuum chamber. One of the bellows is located on top and the other on the bottom of the vacuum chamber. The chief function of the bellows is to support the vacuum while allowing the pull rods to move as the specimen elongates or creeps. Also, the bellows have electric feedthroughs located on them. The lower bellows has three thermocouple feedthroughs on it, while the upper bellows has one vacuum gage feedthrough.

Cross chamber. The cross chamber is located above the lower bellows. The purpose of the cross chamber is to house the main vacuum valve that connects the roughing pump to the chamber. The cross chamber has two face plates on each side. One of the face plates has six electric feedthoughs for the transducer leads and two liquid feedthoughs for the cooling system. The exterior of the cross chamber has a cooling system also to protect the sensitive o-rings.

Main chamber. The main chamber is the part that most resembles an vacuum chamber. It is four feet in length and has an inside diameter of three inches. The main chamber is the only part of the chamber that is directly exposed to the furnace.

Upper chamber. The upper chamber is located just above the main chamber and has an exterior cooling system to protect the o-rings. Thermocouple vacuum tube and gage. The function of the thermocouple vacuum tube and gage is to allow the user to monitor the vacuum condition. The thermocouple vacuum tube is located on the upper bellows and translates the vacuum status to an electrical signal. The thermocouple vacuum gage allows the electrical signal to indicate the vacuum in a millitorr measurement.

Transducer cooling jacket. The transducer cooling jacket purpose is to keep the transducers at a constant temperature. The cooling jacket was made from a single piece of copper tubing and a brass shim stock to create a jacket around the transducers. It was designed to fit within the small tolerances of the vacuum chamber and provide vacuum safe liquid cooling.

Electrical feedthrough. The electrical feedthroughs allows the user to monitor the temperature of the specimen and the transducer inside the vacuum chamber. The electric feedthroughs where assembled and epoxyed by hand. The procedure for the feedthrough assembly can be found in R. Beals thesis 1992.

A.6. VACUUM CREEP EXPERIMENTAL PROCEDURE

The procedure for an vacuum creep experiment is organized into a sequence of assembling and then testing each part of the system. There have been two different designs of the vacuum system. The system originally had the transducers on the outside of the vacuum chamber. However, the established creep facilities do not have this convention and a upgrade of the vacuum system was necessary. The design of the vacuum system was changed to have the transducers on the inside of the vacuum chamber so that they can be connected to the specimen by a extensometer. Thus, the strain measurements The procedure for the vacuum creep experiment is almost exactly like the open-air procedure except that a vacuum chamber is built around the existing system.

The vacuum procedure is as follows:

- 1. Apply antisieze compound same as open-air procedure.
- 2. Assemble the pull-rod system same as open-air procedure.
- 3. Attach the cooling connection to the pull rods same as open-air procedure.
- 4. Clean all the chambers thoroughly with acetone.
- 5. Connect the rough pump to the cross chamber with the foreline hose.
- 6. Place the cross chamber on the lower vacuum support.
- 7. Assemble the main chamber with the upper chamber.

- the placement of the o-rings is very important.

- 8. Put the main chamber and upper chamber on top of the cross chamber
- 8. Place the main chamber on the upper vacuum chamber support.
- 10. Lower a rope through the top of the chamber

- connect the rope to the pull rod system.

- 11. Lift the rope up and hold the pull rod when it reaches the top.
- 12. Attach the upper bellows on the top of the pull rod system.
- 13. Swing the vacuum chamber containing the pull rods into position.
- 14. Screw the pull rod system into the lever arm connection.

Vacuum procedure (cont'd)

15. Attach the cooling jacket for the LCT's

- test to make sure the system does not leak.

16. Attach the transducer leads to the inside of the cross chamber feedthrough.

- label each set of wires

17. Connect the 3 thermocouple feed through to the inside of lower bellows.

- label the thermocouple wire (top,bottom,transducer)

- 18. Connect the bottom of the lower pull rod system to the draw head.
- 19. Attach the cooling connection to the pull rods same as open-air procedure.
- 20. Attach the external cooling connections on the chamber.
- 21. Test all the electrical feedthrough signals and cooling system connections.
- 22. Turn on pump. The procedure to turn on the roughing pump is as follows:
 - a) Close the main vacuum valve on the cross chamber.
 - b) Bakeout the foreline trap.
 - c) Turn on the pump.
 - d) Open the valve.
- 23. Turn on load control same as open-air procedure.
- 24. Turn on the furnace same as open-air procedure.
- 25. Balance the furnace temperature same as open-air procedure.
- 26. Start data collection system same as open-air procedure.
- 27. Start the experiment same as open-air procedure.

A.7. VACUUM SHUT DOWN PROCEDURE

When the vacuum creep experiment is finished creeping the procedure to experimental shut down procedure is listed below:

- Close the main vacuum valve. This is important because once the furnace is turned off the vacuum chamber will experience a pressure difference due to thermal contraction that will cause backstreaming.
- 2. Turn the roughing pump off
- 3. Place the system on load control same as open-air procedure.
- 4. Turn off furnace same as open-air procedure.
- 5. Turn off water same as open-air procedure.
- 6. Turn off controller same as open-air procedure.
- 7. Turn off data collection system same as open-air procedure.
- 8. Bleed vacuum. Carefully turn the vacuum bleeding screw, located on the cross chamber, until the vacuum thermocouple gage starts to move. The chamber should be bled slowly as to protect the sensitive o-rings.
- 9. Disconnect the lower pull rod from the draw head attachment.
- 10. Detach exterior, transducer and pull rod cooling systems.
- 11. Remove pull rod system from the lever arm connection.
- 12. Swing the vacuum chamber back out from the creep frame.
- 13.While holding the top of the pull rod system remove the upper bellows.
- 14. Attach a rope to the top of the pull rod system.

Vacuum shut down procedure (cont'd)

15. Lower the rope and pull rod system

16. Disassemble the pull rod system and remove the specimen.

A.8. RECURRENT PROBLEMS

The previous procedures are very involved, time consuming and problematic. The following are several of the problems that we have encountered in our experiments, and the solutions we used to solve them.

Unload Jack. Before creep experiments commence, the jack must be completely unloaded so that the jack will not bottom out during the experiment. Therefore, the draw head should be used during the heat up process to accommodate any thermal expansions that occur during heat up rather than the jack. Similarly the draw head needs to be in the up position in order to not bottom out during the heat up stage.

Adjust transducers. Another problem is the transducers need to be adjusted so the output signal is between -10 and 10 volts. Because the value cannot be below negative ten and register on the data acquisition system.

Water flow. Water flow is a problem to be aware of. If the water flow stops critical thermal damage could result on the equipment. The best way to tell the water flow through the cooling tube network is by feeling the copper and plastic tubing. A water leak in the vacuum liquid feedthoughs is very dangerous. At high temperatures, if there is a water leak in the main chamber this will lead to a steam bomb effect and will serious

damage the equipment.

Electrical noise & signal drift. The electrical noise or interference in the data collection system has not been identified. However, the signal distortion is small and insignificant. Note: all the electrical systems run on through the same ground and have surge protectors.

APPENDIX II

TABLE OF DATA ON CREEP OF TIAI

COMP.	MICROSTRUCTURE/COND	T/C	TEMP (°C)	σ (MPa)	ч	Q (kJ/mol)	REF
Ti-48Al-2Cr	I/M Nearly lamellar	Т	700	400	14.3	630	∞
	(d = 200mm)		800	400	12.0	630	
			006	400	8.8	630	

COMP.	MICROSTRUCTURE/COND	T/C	TEMP (°C)	σ (MPa)	u	Q (kJ/mol)	REF
Tī-54Al	Self-diffusion of Ti ⁴⁴ isotope		881 - 1400			291	32
Ti-48A1	as-cast: lamellar with strong	ပ	850	300	5.0	269	47
	casting texture	υ	800	330	5.9	460	
	upset forged: equiaxed grains	υ	850	100	5.0	400	
	d = 8-10 µm homogeneous	ပ	800	330	4.3	460	
Ti-48AI-2Cr-2Nb	Duplex	F	705	103	3.0	300	25
	1350°C/.5hr/1350°C/10hr/GFC	Н	815	138	7.0	400	
Ti-48Al-1Nb	equiaxed, $d = 15-50 \ \mu m$	F	704	137.8	4.95	317.5	37
		F	850	206.8	4.95	341	
		F	704	100	5.0	326.4	
		F	800	100	4.9	326.4	
		F	850	100	4.46	326.4	
Ti-48.7AI-2.2W			200 - 900	173 - 345	4.5	400	40
		-	750 - 900	172.4	5.5	370	
Ti-50.3Al			700 - 850	206 - 241	4.0	300	
Ti-53Al-1Nb			832 - 900	69 - 103	6.0	560	

COMP.	MICROSTRUCTURE/COND	T/C	TEMP (°C)	σ (MPa)	u	Q (kJ/mol)	REF
Ti-53.4AI	equiaxed γ , d = 32,90,170 μ m Ar	c	677 - 927	80 - 400	8.0(lowa)	600 (low J)	26, 27
		υ	677 - 927	80 - 400	4.5(higho)	360 (high a)	28, 29
							30
Ti-51.5Al	equiaxed γ , d = 22,32,40 μ m Ar	υ	677 - 927	80 - 400	7.4(low J)	427(low J)	
		C	677 - 927	80 - 400	4.8(high 0)	380(high J)	
Ti-S0AI	equiaxed γ , d = 32,90,170 μ m Ar	C	<i>11 - 927</i>	80	7.1(low g)	460(low J)	
		C	677 - 927	400	4.6(high 0)	350(high v)	
Ti-54Al	$T > 800^{\circ}C$ no twinning	C	750	30 - 200	4.6		35
	T< 800°C twinning	υ	006	30 - 200	3.7		
	no change in (de/dt) due to twin						-
Ti-48A1-2Nb-2Cr	ingot + HIPed + homogenized	Т	540 - 870	105 - 550		320	23
	+ extruded - stress rupture						
Ti-50AI	P/M + extrusion + heat treatment		700 - 950	103 - 241	4.0(T=850)	300	6
Ti-50Al-2.2W	P/M + extrusion + heat treatment		700 - 950	138 - 310	5.5(T=850)	370	
Ti-47Al-1Cr-1V-2.5Nb	cast + HIP + extrusion (FL)	Т	006	138 - 276	5.8		36
Ti-47AI	P/M equaxed $d = 4 \ \mu m$, $p = 2$	Т	006 - 009	34 - 138	1.9	340 <u>+</u> 31	48
Ti-47Al + 4vol%TiB2	Ingot equiaxed d = 8-18 µm, p = 2	Т	006 - 009	34 - 138	3.0 - 3.5	340 ± 31	
					1		

	1	6	1
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COMP.	MICROSTRUCTURE/COND	T/C	TEMP (°C)	σ (MPa)	U	Q (kJ/mol)	REF
Ti-46Al-2V + 6.6vol%TiB ₂	extruded + 1200°C/16hr + 1000°C/5hr		760 760 - 840	69 103(760)	1.9 1.9(760)	330	49
			760 760 760	138 172 241	3.5 3.5 3.5		
Ti-48Al + 7vol%TiB2 (TiB2 + Ti2AlN)	as extruded		760	38 - 276	2.4		
Ті-43АІ	equiaxed, d = 5 µm, superplastic	T/C	1000 - 1100	35 - 400	2 (low σ) 4 (hieh σ)	380-390	50
TI-49.5Al-2.5Nb-1.1Mn	equiaxed g, d = 125 mm + 10-15% lamellar g/a2 cast + HIPed	F	850 - 1377	75 - 200		411	41
	equiaxed γ , d = 35 μ m extruded + heat treated	F	850 - 1377	75 - 200		411	

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COMP.	MICROSTRUCTURE/COND	T/C	TEMP (°C)	σ (MPa)	L	Q (kJ/mol)	REF
Ti-48A1	extruded + heat treatment	Ч	768	105	4.8	278	51
	900°C,24hr (NL)	Н	815	207	4.8	278	
	extruded + heat treatment	Ч	768	105	4.6	350	
	1350°C,.5hr + 900°C,6hr	Т	815	207	4.6	350	
Ti-24Al-11Nb	as extruded, coarse equiaxed	F	593	137.9	5.1	196	52
	$d = 10 \ \mu m$ & heat treated	Т	704	206.9	5.4	352	
	widemanstatten w/in α_2 grains	-	760	275.8	5.6	352	
	d = 15 µm						
	two phase, dislocation climb	F	650 - 760	69-172	4.3	260	
Ti-47AI	P/M + 6vol% TiB2 air		800	34 - 138	1.9		53
	I/M + 6vol% TiB2		800	34 - 138	3.0 - 3.5		
			006 - 009	69		340	
Ti-48AI-2Cr	I/M (DP)	F	800	330	4.3	460	54

COMP.	MICROSTRUCTURE/COND	T/C	TEMP (°C)	σ (MPa)	u	Q (kJ/mol)	REF
Ti-48Al-2Cr-2Nb	I/M Duplex	Т	700	200	12.5	513	∞
	(11111C7 = D)		800	200	7.2	513	
			006	200	4.7	513	
			700	400	12.5	630	
			800	400	7.2	630	
			006	400	4.7	630	
Ti-48A1-2Cr	I/M Nearly lamellar		700	200	2.4	292	
	(UIIU007 = D)		800	200	2.3	292	
			006	200	3.5	292	
Ti-48A1-2Cr	I/M Nearly lamellar	Ц	700	200	2.4	292	
	(111111007 = D)		800	200	2.3	292	
			006	200	3.5	292	

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