INFORMATION TO USERS

This manuscript has been reproduced from the microfilm master. UMI films the text directly from the original or copy submitted. Thus, some thesis and dissertation copies are in typewriter face, while others may be from any type of computer printer.

The quality of this reproduction is dependent upon the quality of the copy submitted. Broken or indistinct print, colored or poor quality illustrations and photographs, print bleedthrough, substandard margins, and improper alignment can adversely affect reproduction.

In the unlikely event that the author did not send UMI a complete manuscript and there are missing pages, these will be noted. Also, if unauthorized copyright material had to be removed, a note will indicate the deletion.

Oversize materials (e.g., maps, drawings, charts) are reproduced by sectioning the original, beginning at the upper left-hand corner and continuing from left to right in equal sections with small overlaps. Each original is also photographed in one exposure and is included in reduced form at the back of the book.

Photographs included in the original manuscript have been reproduced xerographically in this copy. Higher quality 6" x 9" black and white photographic prints are available for any photographs or illustrations appearing in this copy for an additional charge. Contact UMI directly to order.

UMI

A Bell & Howell Information Company
300 North Zeeb Road, Ann Arbor MI 48106-1346 USA
313/761-4700 800/521-0600

INVESTIGATION OF PRIMARY CREEP IN NEAR γ -TITANIUM ALUMINIDE

by

DAVID CHRISTOPHER DUDZINSKI, B.ENG.

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

Master of Engineering

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

Carleton University
Ottawa, Ontario
September 1997

© 1997
David Dudzinski

National Library
of Canada

Acquisitions and
Bibliographic Services

395 Wellington Street
Ottawa ON K1A 0N4
Canada

Bibliothèque nationale
du Canada

Acquisitions et
services bibliographiques

395, rue Wellington
Ottawa ON K1A 0N4
Canada

Your file Votre référence

Our file Notre référence

The author has granted a non-exclusive licence allowing the National Library of Canada to reproduce, loan, distribute or sell copies of this thesis in microform, paper or electronic formats.

The author retains ownership of the copyright in this thesis. Neither the thesis nor substantial extracts from it may be printed or otherwise reproduced without the author's permission.

L'auteur a accordé une licence non exclusive permettant à la Bibliothèque nationale du Canada de reproduire, prêter, distribuer ou vendre des copies de cette thèse sous la forme de microfiche/film, de reproduction sur papier ou sur format électronique.

L'auteur conserve la propriété du droit d'auteur qui protège cette thèse. Ni la thèse ni des extraits substantiels de celle-ci ne doivent être imprimés ou autrement reproduits sans son autorisation.

0-612-26993-0

Carleton University
Ottawa, Canada K1S 5J7

1942-1992

Thesis contains black and white and/or coloured graphs/tables/photographs which when microfilmed may lose their significance. The hardcopy of the thesis is available upon request from Carleton University Library.

The undersigned hereby recommend to the
Faculty of Graduate Studies and Research acceptance of the thesis,

INVESTIGATION OF PRIMARY CREEP
IN NEAR γ -TITANIUM ALUMINIDE

by

DAVID CHRISTOPHER DUDZINSKI, B.ENG.

in partial fulfillment of the requirements
for the degree of Master of Engineering

Dr. R. Bell

Chair, Department of Mechanical and
Aerospace Engineering

Dr. J. Beddoes, Thesis Supervisor

Carleton University
1997

ABSTRACT

A review is given of the relevant physical metallurgy associated with creep of engineering alloys, as well as an overview of the technology related to near γ -TiAl intermetallics.

The primary creep behaviour at 760°C and an applied stress of 172 MPa and 240 MPa is presented for a binary Ti-48at%Al intermetallic. Several microstructures were examined including a duplex, and three fully lamellar conditions. The monotonic creep results indicate that, at both stresses, the fully lamellar microstructures exhibit better primary creep resistance than the duplex structure. A further increase in the primary creep resistance occurs by reducing the lamellar interface spacing.

After primary creep deformation, dislocation loops emanating from the lamellar interfaces, as well as bowed dislocation segments spanning the γ lamellae exist, suggesting that the primary creep behaviour is dependent on both the emission of dislocations from lamellar interfaces, and the combined glide and climb of such dislocations in the γ lamellae.

Results from anelastic creep tests, which followed near total stress reduction during primary creep, indicate the anelastic response occurs in two stages. The recoverable anelastic strain increases as the applied primary creep increases.

ACKNOWLEDGEMENTS

I would like to express my deepest appreciation and utmost respect for Dr. J. Beddoes. His continued guidance, insightful conversations, and dedicated nature made this work not only enjoyable, but an exciting learning experience.

I wish to thank Carleton University and the Natural Science and Engineering Research Council of Canada for financial assistance. I am very grateful to Dr. L. Zhao and Dr. P. Au for their encouraging discussions and assistance throughout my research. As well, the support of many colleagues and friends, D. Morphy, E. Galasso, and J. Alo was greatly appreciated.

Most importantly, I would like to thank my family, in particular (but not limited to) my parents, Kenneth and Heather, my sisters, Karen and Rebecca, and my Nana for their support and encouragement throughout my studies. Above all, I would like to thank my future wife and best friend, Jennifer.

Two handwritten signatures are shown. The top signature is written over a horizontal line and appears to be 'Iain'. The bottom signature is written over a horizontal line and appears to be 'Jennifer'.

TABLE OF CONTENTS

<u>Item</u>	<u>page</u>
Acceptance Sheet	ii
Abstract	iii
Acknowledgments.....	iv
Table of Contents.....	v
List of Tables	vii
List of Figures	viii
Nomenclature.....	xi
 CHAPTER 1 - Introduction	 1
 CHAPTER 2 - Literature Review	 7
2.1 Background Review	7
2.1.1 Geometry and Movement of Dislocations	7
2.1.1.1 Dislocation Sources	9
2.1.2 Strain Response of Metallic Structures to an Applied Stress	11
2.1.3 Creep of Metallic Crystalline Structures	13
2.1.3.1 Anelastic Creep Strain	14
2.1.4 Creep Deformation Mechanisms	15
2.1.4.1 Diffusional Creep	16
2.1.4.2 Dislocation Creep	17
2.1.4.3 Grain Boundary Sliding	17
2.1.5 Creep Testing	18
2.1.5.1 Anelastic Creep Test	19
2.2 Near γ -TiAl Intermetallics	21
2.2.1 Crystal Structure of γ -TiAl	21
2.2.2 Near γ -TiAl Microstructures	22
2.2.3 Mechanical Properties of Near γ -TiAl Intermetallics	27
2.2.3.1 Tensile Strength and Ductility	27
2.2.3.2 Fracture Toughness	29
2.2.3.3 Creep of Near γ -TiAl Intermetallics	29
2.2.4 Microstructural Features Affecting Creep of Near γ -TiAl Intermetallics..	31
2.2.4.1 Grain Size	31
2.2.4.2 Lamellar Spacing	32
2.2.4.3 Grain Boundary Morphology	33
2.2.4.4 Single Phase γ Grains	33

2.2.4.5 Lamellar Orientation	34
2.2.4.6 Lamellar Interfaces	35
2.3 Summary	36
CHAPTER 3 - Thesis Objective and Scope	37
CHAPTER 4 - Materials and Experimental Procedure	39
4.1 Materials	39
4.2 Material Preparation	40
4.3 Heat Treatment	42
4.4 Creep Testing Procedure	43
4.5 Microstructural Analysis	45
CHAPTER 5 - Results	46
5.1 Initial Microstructures	46
5.2 Mechanical Test Results	47
5.2.1 Anelastic Analysis and Results	49
5.3 Deformed and Anelastic Recovered Microstructures	50
5.3.1 Deformed Microstructures	51
5.3.2 Anelastic Recovered Microstructures	51
CHAPTER 6 - Discussion	52
CHAPTER 7 - Summary and Conclusions	58
7.1 Suggested Future Work	59
References	61
Tables	66
Figures	73
APPENDICES	112
Appendix A - LVDT Sensitivity Graphs	113
Appendix B - Fortran Source Code for Data Acquisition System.....	116

LIST OF TABLES

- Table I - Comparison of the Mechanical Properties of Near γ -TiAl with Titanium Alloys and Superalloys^[8].
- Table II - Summary of Microstructural Features and Monotonic Creep Results at 240 MPa and 760°C^[12].
- Table III - Chemical Composition of Testing Material -- Ti-48Al.
- Table IV - Summary of Heat Treatments.
- Table V - Summary of 760°C Creep Test Results.
- Table VI - Anelastic Strain Results After 0.2% Creep Strain at 760°C and 172 MPa.
- Table VII - Anelastic Strain Results After 0.4% Creep Strain at 760°C and 240 MPa.

LIST OF FIGURES

- Figure 1: Manufactured near γ -TiAl exhaust valves developed by Ford Motor Company^[3].
- Figure 2: Full set of 5th stage low pressure turbine blades fabricated from Ti-48Al-2Cr-2Nb tested by General Electric in a CF6-80C engine^[5].
- Figure 3: Central portion of the Ti-Al phase diagram^[6].
- Figure 4: Schematic of creep curve.
- Figure 5: Dislocation schematics in a simple cubic structure;
(a) edge dislocation^[14], and (b) screw dislocation^[16].
- Figure 6: Micrograph of dislocation pileup on two systems against a grain boundary^[14].
- Figure 7: Schematic of Frank-Read dislocation source^[14, 17];
(a) pinned dislocation segment,
(b) dislocation segment bowing out of its glide plane,
(c,d) unstable segment beginning to bend around itself,
(e) completion of closed loop, and
(f) original line segment with a new dislocation loop.
- Figure 8: Micrograph of a Frank-Read dislocation source^[14, 17].
- Figure 9: Schematic representation of double cross-slip mechanism^[14].
- Figure 10: Schematic representation of strain response to stress;
(a) elastic, (b) viscous, (c) anelastic, and (d) plastic.
- Figure 11: Graph defining the relaxation time constant^[15].
- Figure 12: Schematic strain transient following a reduction in the applied stress^[21];
(a) instantaneous elastic contraction, (b) time dependent backflow,
(c) region of zero creep rate, (d) constant structure forward creep rate, and
(e) new steady state.
- Figure 13: Schematic of vacancy diffusion at high temperatures (Nabarro-Herring creep)^[14].
- Figure 14: Grain boundary sliding (GBS) creates voids (gray area) which are concurrently filled by diffusion creep or conversely diffusion creep causes GBS^[25].
- Figure 15: Schematic of a constant load creep frame^[26].
- Figure 16: Photograph of extensometer.

- Figure 17: a) Schematic plot of incubation periods associated with a stress decrement test^[12] and,
b) schematic of cumulative incubation periods with cumulative stress reductions^[12].
- Figure 18: Anelastic strain rate response for Al^[21].
- Figure 19: Atomic arrangements of titanium aluminide intermetallic crystal structures^[32];
(a) γ – TiAl with face centered tetragonal structure, and
(b) α_2 – Ti₃Al with hexagonal closed-packed structure.
- Figure 20: Micrograph of typical microstructures^[11];
(a) duplex, (b) near gamma, (c) nearly lamellar, and (d) fully lamellar.
- Figure 21: Typical heat treated microstructures.
- Figure 22: Grain boundary morphology of TiAl after 1375°C heat treatments^[7].
- Figure 23: Typical duplex microstructure.
- Figure 24: Scanning electron microscope-backscattered electron image indicating four orientations of α in γ .
- Figure 25: Typical FGFL microstructure.
- Figure 26: Effect of microstructure on grain size (GS) and mechanical properties including: fracture toughness, tensile strength and elongation (EI), impact resistance (IR), and creep resistance (CR). Creep resistance was defined as the reciprocal of the secondary creep strain rate^[33].
- Figure 27: Monotonic creep test results for Ti-48Al at 760°C and 240 MPa^[12].
- Figure 28: Properties of lamellar single crystal as a function of lamellar orientation to the applied load^[43];
(a) yield strength, and (b) tensile elongation.
- Figure 29: Schematic representation of the crucible refractory free process for titanium powders.
- Figure 30: Photograph showing progression of TiAl powder to the final creep specimen.
- Figure 31: Photograph showing hydraulically operated crimping tool.
- Figure 32: Photograph of vacuum system showing:
(a) roughing pump, (b) diffusion pump, (c) filled tube being evacuated, and (d) vertical furnace for degassing.
- Figure 33: Photograph of inert gas chamber used to fill stainless steel tubes with TiAl powder.
- Figure 34: Schematic representation of the hot isostatic press.

- Figure 35: Technical drawing of the creep specimen.
- Figure 36: Photograph of HIP'ed microstructure.
- Figure 37: Typical as heat-treated microstructures.
- Figure 38: TEM micrographs of lamellar regions of duplex, FLn, and FLw in the as heat-treated condition.
- Figure 39: Comparison of creep results with and without elastic strain portion corresponding to initial loading.
- Figure 40: Response of FLn condition at 760°C and 240 MPa initial stress.
- Figure 41: Response of FLn, FLw, duplex, and FGFL conditions at 760°C and 172 MPa initial stress.
- Figure 42: Response of FLn, FLw, duplex, and FGFL conditions at 760°C and 240 MPa initial stress.
- Figure 43: Strain Response of FLn condition at 760°C and 240 MPa initial stress.
- Figure 44: Anelastic response following stress reduction at 760°C and 240 MPa initial stress for FLn condition.
- Figure 45: Anelastic response of FLn, FLw, duplex, and FGFL conditions after 0.2% creep strain at 760°C and 172 MPa initial stress.
- Figure 46: Anelastic response of FLn, FLw, duplex, and FGFL conditions after 0.4% creep strain at 760°C and 240 MPa initial stress.
- Figure 47: Typical TEM micrographs of FLn and FLw microstructures after 0.2% deformation at 172 MPa;
(a) FLn, and (b) FLw.
- Figure 48: Typical TEM micrographs of FLn and FLw microstructures after 0.4% deformation at 240 MPa;
(a) FLn, and (b) FLw.
- Figure 49: Typical TEM micrographs of FLn and FLw microstructures after recovery;
(a) FLn, and (b) FLw.

NOMENCLATURE

$^{\circ}\text{C}$	- degrees centigrade
α	- Ti phase (disordered hexagonal close packed)
α_2	- Ti_3Al phase (ordered hexagonal closed-packed)
β	- Ti phase (body centered cubic)
γ	- TiAl phase (ordered face-centered tetragonal)
ϵ_{an}	- anelastic strain
ϵ_e	- elastic strain
ϵ_f	- fracture strain
ϵ_p	- plastic strain
ϵ_R	- total amount of strain
ϵ_U	- immediate elastic contraction
$\dot{\epsilon}$	- strain rate
$\dot{\epsilon}_{\text{min}}$	- minimum strain rate
$\dot{\epsilon}_s$	- steady state strain rate
ρ	- dislocation density
σ	- applied stress
τ	- relaxation time
v	- dislocation velocity
μm	- microns
$\gamma\text{-TiAl}$	- near γ -titanium aluminide
a	- fraction of the total strain which lags behind application of the load
A	- material constant
AC	- air cooled
Al	- aluminum
atm	- atmosphere
b	- Burger's vector
BCC	- body centred cubic
d	- grain diameter
D	- diffusivity
DBTT	- ductile-to-brittle transition temperature
D_{gb}	- diffusivity along the grain boundary
D_v	- volume diffusivity through the grain
E	- modulus of elasticity
FAC	- forced air cooled
FC	- furnace cooled
FGFL	- fine grain fully lamellar
FL	- fully lamellar
FLn	- fully lamellar narrow

FLw	- fully lamellar wide
G	- shear modulus
HCP	- hexagonal closed packed
Hg	- mercury
HIP	- hot isostatic press
k	- Boltzman's constant
kg	- kilogram
K _{IC}	- plane-strain fracture toughness
L	- lamellar morphology
LVDT	- linear variable differential transformer
m	- meter
mbar	- millibar
mm	- millimeter
MPa	- Mega Pascals
NG	- near gamma
NL	- nearly lamellar
nm	- nanometer
R	- universal gas constant
r	- dislocation radius of curvature
t	- time
t _U	- time of unloading
t _f	- fracture time
T	- absolute temperature
TGA	- titanium gas atomizer
Ti	- titanium
TIG	- tungsten inert gas
T _α	- alpha transus temperature
T _m	- absolute melting temperature
TiAl	- titanium aluminide

CHAPTER 1

INTRODUCTION

In the last two decades there has been an increase in the active research and development of near γ -titanium aluminide (γ -TiAl) intermetallics for numerous reasons. Foremost among these is the potential of near γ -TiAl as a lightweight, high-temperature material for automotive and aerospace applications. This potential stems from the material's low density, high modulus of elasticity, and high temperature properties including good oxidation resistance.

The unique properties of near γ -TiAl intermetallics are useful for automotive power-train applications^[1]. Recent trends to near stoichiometric fuel:air mixtures and higher engine speeds increase the valve temperatures^[2]. Near γ -TiAl intermetallics would allow the valves to operate at higher temperatures^[2]. The low density of near γ -TiAl also provides significant benefits to the engine including higher fuel economy and reduced noise and vibration^[2,3]. For example, a reduction in the weight of engine exhaust valves^[1-4] would mean that lower spring loads are required to close these valves. In turn, the engine is required to produce less energy to open the valves. Other potential automotive components include turbocharger rotors^[1,4], and connecting

rods^[1]. Both General Motors and Ford have tested near γ -TiAl for several years. Figure 1^[3] shows an example of the results of this development effort; near γ -TiAl exhaust valves manufactured by the Ford Motor Company.

Aerospace applications of near γ -TiAl include gas turbine engine parts such as high pressure compressor or low pressure turbine blades, as well as structural components such as turbine frame and fan sections. The low density of near γ -TiAl provides an advantage in reduced weight for these components. Application trials of near γ -TiAl in the aerospace industry have been successfully completed. One such example includes a full set of 5th stage low pressure turbine blades, investment cast from Ti-48%Al-2%Cr-2%Nb*, as shown in Figure 2^[5]. The 5th stage section was tested by General Electric in a CF6-80C engine. The tests were successful with no problems related to the material used and showed no significant corrosion or oxidation.

Near- γ titanium aluminides are intermetallic compounds. An intermetallic forms when a specific solute element is added to a host metal resulting in an ordered crystal structure that differs considerably from the structure of each constituent. In the binary titanium-aluminum system, γ -TiAl intermetallics of engineering interest typically range from 45 to 50 atomic percent aluminum. The equilibrium Ti-Al phase diagram is shown in Figure 3^[6]. From this it is evident that equilibrium compositions of near γ -TiAl will

* All compositions expressed as atomic percent unless otherwise indicated.

contain a large volume fraction of γ and a minor volume fraction of α_2 . The temperature which separates the $\alpha+\gamma$ and the α phase fields, the alpha-transus temperature (T_α), is approximately $1370\pm 5^\circ\text{C}$ ^[7] for an aluminum content of 48%. It is also clear that appropriate heat treatments in the single phase α or two phase $\alpha+\gamma$ or $\alpha_2+\gamma$ phase fields will produce different microstructures, thereby modifying the mechanical properties. The microstructure-property relationships will be discussed in the following chapter.

A comparison between the key mechanical properties of superalloys, titanium alloys, and near γ -TiAl is presented in Table I^[8]. One significant advantage of near γ -TiAl is that it has a low density which is approximately half that of current nickel based superalloys. As well, the ordered structure of intermetallics, including near γ -TiAl, significantly reduces diffusion rates. This leads to a higher temperature capability compared to titanium alloys, as shown in Table I. However, there exist some shortcomings of key properties required for application including:

- insufficient oxidation resistance above 800°C ,
- relatively poor high temperature strengths for application at 1000°C , and
- difficulty in producing a microstructure that concurrently has good tensile properties, fracture toughness, and creep resistance which results in a poor combination of mechanical properties^[9].

The last point is of particular importance. As will be explained in section 2.2.3, near γ -TiAl intermetallics are unique in that tensile ductility and fracture toughness are

inversely related. For many applications however, creep resistance is the property that limits application temperature and/or service life.

Creep is the time dependent strain resulting from a continually applied stress. Creep occurs in various environments ranging from the earth's plate tectonics to advanced aerospace gas turbine engines. A typical creep curve for a metallic material is shown schematically in Figure 4 and exhibits three distinct stages: primary, secondary, and tertiary. During primary creep the strain rate decreases. This is followed by a nearly constant strain rate in the secondary stage. Finally in the tertiary stage, the strain rate increases leading to fracture. The shape of the creep curve is dependent on a number of factors including the material chemical composition, operating conditions, and microstructure.

For many gas turbine applications, primary creep is of considerable importance. To retain satisfactory operation of a turbine, component dimensional changes must be minimal. Therefore many components are considered to have failed after strain levels corresponding to primary creep deformation have occurred. As an example, the allowable creep deformation of high pressure compressor vanes is typically only 0.2 percent elongation after 500 hours at 760°C and a stress of 69 MPa^[10].

For near γ -TiAl, primary creep results in significant deformation which may exceed the projected design limit for acceptable strain in a component^[11]. Despite this initial

deformation, the primary creep behaviour of near γ -TiAl has not been extensively studied compared to secondary creep, which accounts for the majority of creep life^[11]. However, to fully understand the deformation occurring during secondary and tertiary creep, it is necessary to determine the extent and mechanisms of primary creep. It is also important to identify the microstructural evolution during these stages. Given the significance of primary creep, both from the viewpoint of gas turbine applications and understanding subsequent creep deformation, the objective of this thesis is to investigate the primary creep behaviour of a near γ -TiAl intermetallic. In particular, the primary creep behaviour as a function of microstructure and stress level is to be determined. Further insight can be obtained by monitoring the anelastic strain response after a stress drop in the primary creep region.

The material used for this thesis is a binary near γ -TiAl intermetallic containing 48 atomic percent aluminum (Ti-48Al). This composition was chosen for two important reasons. Firstly, a *binary* near γ -TiAl was selected to avoid the presence of tertiary elements in the composition which would unduly complicate the results. This would make the determination of the fundamental aspects of the primary creep response more difficult. Secondly, previous research on this particular composition provides required baseline creep data^[12,13].

In the following chapter, a review of the current state of near γ -TiAl technology is presented. Chapter 3 details the specific objectives of this thesis, while the materials and

experimental procedure are presented in Chapter 4. The results and discussion are presented in Chapters 5 and 6 respectively. In the final chapter, a summary and conclusions are given and suggestions for future work made.

As will become apparent in the subsequent chapters, there appears to be some disagreement in the literature regarding the primary creep resistance of the fully lamellar condition. However, one of the most important results of this thesis is the clear conclusion that near γ -TiAl intermetallics with a fully lamellar microstructure exhibit significantly improved primary creep resistance compared to the duplex condition. The improved primary creep resistance is consistent with the already well established lower strain rates of the fully lamellar microstructure during the secondary stage of creep. Therefore, for near γ -TiAl intermetallics, the fully lamellar microstructural condition offers improved creep behaviour over the entire range of strains of importance for many gas turbine applications.

CHAPTER 2

LITERATURE REVIEW

This section has been separated into two parts. The first is a review of the physical metallurgical topics of relevance to the current thesis. The second part presents an overview of the technology specifically related to near γ -TiAl intermetallics.

2.1 Background Review

The following is a review of the geometry and movement of dislocations and dislocation sources. In keeping with the emphasis and nature of this thesis an examination of the following topics will be presented; the strain response of metallic structures to an applied stress, the creep of metallic crystalline structures, creep deformation mechanisms, and creep testing.

2.1.1 GEOMETRY AND MOVEMENT OF DISLOCATIONS

A dislocation is a crystallographic line defect that defines the boundary between slipped and unslipped portions of the crystal^[14]. Dislocations can be resolved into edge components and screw components, as shown in Figure 5a^[15] and 5b^[16] respectively. The difference between edge and screw dislocations may be understood by considering

the Burger's circuit. The Burger's circuit is a series of interatomic steps along lattice directions that generate a closed loop about any location in the crystal lattice. In a perfect lattice, the Burger's circuit will return to its starting position after an equal and opposite number of interatomic steps in the horizontal and vertical directions. Dislocations in the lattice prevent the Burger's circuit from closing. The interatomic step needed to close the Burger's circuit is called the Burger's vector and represents both the magnitude and direction of slip associated with the dislocation. The edge dislocation (Figure 5a) is defined by the extra half plane of atoms and a Burger's vector oriented *perpendicular* to the dislocation. In comparison, the Burger's circuit about the screw dislocation assumes the shape of a helix, wherein a 360° rotation produces a Burger's vector *parallel* to the dislocation (Figure 5b). The glide movement of a dislocation through a crystal lattice can only occur on a plane that contains the dislocation and Burger's vector. Edge dislocations are therefore confined to a glide on a single plane. This differs for screw dislocations for which there are many glide planes. Mixed dislocations, such as dislocation loops, can have a combination of both edge and screw properties. Dislocations can terminate at a free surface or grain boundary but never within the crystal^[14]. Thus dislocations must form closed loops, networks with branches that terminate at a surface^[14], or have each end of the dislocation terminate at a surface.

When a queue of dislocations is forced against some obstacle by an applied stress, it is called a dislocation pileup. An example in Figure 6^[14] shows the pileup of edge dislocations against a grain boundary which is blocking the motion of the leading

dislocation. This dislocation is acted upon not only by the applied stress, but also by the interaction force with the other dislocations in the pileup^[17]. The lagging dislocation is also acted upon by the applied stress as well as the interaction force with the other dislocations. However for the lagging dislocation, the stress resulting from dislocation interactions is in the opposite direction compared to that of the leading dislocation. In this manner it is possible for an internal stress to act in the opposite direction of an externally applied stress. Internal stresses that act in the opposite direction to the applied stress are referred to as back stresses.

2.1.1.1 DISLOCATION SOURCES

The mobile dislocation density is a major factor that can effect the mechanical properties of a material, including creep. During periods of stress change, it is well known that the dislocation density will change proportionately to a value characteristic of the new strain rate. Following the Orowan relationship for strain, the strain rate can be written as^[18]:

$$\dot{\epsilon} = \rho b v \quad \dots [1]$$

where ρ is the dislocation density, b is the Burger's vector, and v is the dislocation velocity. Well annealed pure metals contain a dislocation density of about 10^6 cm/cm^3 . During the early stages of deformation (up to strains of approximately ten percent) the dislocation density of these metals increases to a typical value of 10^{10} cm/cm^3 ^[17]. Although there are a number of possible dislocation generation mechanisms which have

been experimentally verified, it is presently not possible to determine which one is dominant^[17]. The following discussion on dislocation generation briefly introduces dislocation sources that may occur during creep deformation.

One type of dislocation multiplication mechanism, which is widely observed in many metallic structures is the Frank-Read source^[14]. A schematic of this source is shown in Figure 7^[14]. In Figure 7a a dislocation segment is pinned by foreign atoms, particles, or by interaction with other dislocations. When a shear stress is applied the dislocation will bow out as shown in Figure 7b. An approximation of an applied shear stress required to bow out a dislocation segment is given by^[14]:

$$\tau \approx \frac{Gb}{r} \quad \dots[2]$$

where G is the shear modulus, b is the Burger's vector, and r is the radius of curvature of the dislocation. Dislocation bowing will increase with increasing applied stress, while the radius of curvature decreases to the point where r equals half the length of the pinned segment. The segment then becomes unstable and will start to bend around itself (Figure 7c and 7d). The dislocation segment then connects forming a loop (Figure 7e) because the two regions bending around itself (Figure 7d) correspond to two screw dislocations of opposite sign. Finally the loop becomes circular and the remaining dislocation segment will become straight to minimize their length (Figure 7f). With further stress application, the loop can move out radially and the dislocation segment can bow out again to form additional loops as shown in Figure 8^[14, 17]. Theoretically, an

infinite amount of dislocations can be generated. However the source in reality is shut down by back stresses produced by the pileup of dislocations against unyielding obstacles^[14].

A closely related mechanism to the Frank-Read source is the double cross-slip mechanism shown in Figure 9^[14]. A screw dislocation will cross-slip onto a plane with a lower resolved-shear-stress when an obstacle is encountered. The cross-slipped segment then cross-slips back onto the original glide plane after the dislocation has completely passed the obstacle. The screw dislocation that has cross-slipped twice can now function as a Frank-Read source^[14, 17].

2.1.2 STRAIN RESPONSE OF METALLIC STRUCTURES TO AN APPLIED STRESS

Four different types of strain response can occur to an applied stress and are shown schematically in Figure 10. Firstly, elastic strain is characterized by the appearance and disappearance of strain with the application and removal of stress respectively (Figure 10a). Elastic strain (ϵ_{el}) is independent of microstructure and can be determined according to Hooke's Law^[14]:

$$\epsilon_{el} = \frac{\sigma}{E} \quad \dots[3]$$

where σ is the applied stress and E is the modulus of elasticity.

A second type of strain is viscous deformation, for which the strain rate is proportional to the applied stress (Figure 10b). The time dependent component of strain which is recoverable, is often referred to as anelastic strain (ϵ_{an}). That is, a maximum strain value is obtained after the maximum value of stress, and yet some portion of the strain can be recovered, over a period of time, after the stress is removed^[19] (Figure 10c). The time dependent component of strain can be described by a single parameter representing the relaxation time, τ ^[15]. To understand the meaning of this concept, consider a specimen which has the stress suddenly unloaded at time $t=t_U$ (see Figure 11). The specimen undergoes an immediate elastic contraction (ϵ_U). With the passage of time, the specimen slowly approaches the initial strain-free state^[15]. The time-dependent component of strain is often approximated as an exponential function of time. If 'a' is the fraction of the total strain which lags behind application of the load, that is,

$$a = \frac{\epsilon_R - \epsilon_U}{\epsilon_R} \quad \dots[4]$$

then the anelastic strain may be expressed by,

$$\epsilon_{an} = a\epsilon_R e^{-[(t-t_U)/\tau]} \quad \dots[5]$$

where ϵ_R is the total amount of strain the specimen endures and τ is a measure of the time required for relaxation or the time required for the time-dependent component of strain to decrease to within $1/e$ of the final value on unloading.

Finally plastic deformation can occur. In this type of deformation, permanent strain is observed with the application of stress. A schematic of plastic strain for metals that work

harden is shown in Figure 10d. In some circumstances more than one type of deformation can occur simultaneously^[19].

2.1.3 CREEP OF METALLIC CRYSTALLINE STRUCTURES

A schematic creep curve is shown in Figure 4 with the three distinctive regions known as the primary, secondary, and tertiary creep regions. The shape of the creep curve is dependent on many factors including applied stress, composition, microstructure, environment, temperature, and time. For example, increasing both temperature and applied stress will cause an earlier onset of the tertiary stage of creep^[20] from that shown in Figure 4.

Before the primary stage, instantaneous elastic strain is observed when the load is applied. Further to this, in the primary region, the metal will harden causing the rate at which the specimen is elongating, or the creep strain rate ($\dot{\epsilon}$), to decrease. This is believed to be the result of the development of stable substructures, which resist dislocation motion and thus decrease the strain rate. A nearly constant $\dot{\epsilon}$ is indicative of secondary creep where the substructure remains unchanged as strain accumulates. This region is often referred to as the steady-state creep regime reflecting the balance between strain hardening and softening processes^[14]. The steady-state creep regime can be very short such that immediately after the primary stage the tertiary stage commences. Therefore, the term minimum strain rate ($\dot{\epsilon}_{\min}$) is frequently used instead of steady-state creep rate. The final or tertiary region of creep, results from an unbalance in the

hardening and softening mechanisms. Microstructural changes such as dynamic recrystallization or microvoid formation contribute to an increasing $\dot{\epsilon}$, which ultimately leads to instability and rupture of the specimen.

2.1.3.1 ANELASTIC CREEP STRAIN

A typical anelastic strain transient for a metal is schematically shown in Figure 12^[21]. After an immediate stress reduction to a lower stress level at time t_0 in region A, an initial instantaneous contraction occurs. This contraction may be entirely elastic and/or may contain an inelastic component of anelastic strain. Region B is characterized by time-dependent backflow (anelastic strain) observed primarily for large stress reductions. The time dependency of the anelastic strain demands an inhomogeneous distribution of stresses within the deforming solid in order to develop the driving force (back stress) necessary for backflow^[21]. For small decrements, region B may be obscured by region A or it may be entirely absent because of the insufficient driving force needed to cause backflow. Observation of a period of zero creep, region C, depends on the resolution of the measuring system. This period is a result of the back stress being equivalent to the applied stress. When the back stress becomes smaller than the applied stress, the sample will again begin to creep at a rate observed in region D. The new creep rate is typically less than that expected for an uninterrupted test at the reduced stress level as a result of the back stress. Eventually, the back stress is reduced and a steady-state creep rate is observed in region E^[21].

The recoverable anelastic strain has been observed to increase during primary creep and saturate as the primary creep strain is exhausted^[21]. Generally for pure metals, the anelastic strain recovered can be several times the elastic contraction associated with the stress change. For more complex engineering alloys, anelastic recovery may only be a fraction of elastic strain^[22]. The annihilation of dislocations after unloading has been suggested^[22] to be one possible explanation for large anelastic strains. The dislocation density is known to vary with changes in applied strain and therefore after unloading, the dislocation density is likely to reduce to a value characteristic of the strain level. By integrating equation 1, the anelastic strain resulting from a dislocation density change may be written as^[22]:

$$\varepsilon_{an} = \rho b x \quad \dots[6]$$

where ρ is the dislocation density, b is the Burger's vector, and x is the average distance dislocations move before annihilation.

2.1.4 CREEP DEFORMATION MECHANISMS

Crystalline solids deform plastically by different, often competing mechanisms^[23]. Several diffusional and dislocation related mechanisms occur during creep resulting in the accumulation of strain. The major mechanisms are summarized in the following sections.

2.1.4.1 DIFFUSIONAL CREEP

Diffusional creep is a low stress mechanism in which vacancy movement occurs. At low temperatures, as first described by Coble, the movement of vacancies is generally from the grain boundary loaded in tension to boundaries loaded in compression^[14]. Concurrently atoms moving in the opposite direction cause elongation of the material. The steady state Coble creep strain rate can be described by^[14]:

$$\dot{\epsilon}_s \approx \frac{50\sigma D_{gb} b^4}{kTd^3} \quad \dots[7]$$

where σ is the applied stress, D_{gb} is the volume diffusivity along the grain boundary, b is the Burger's vector, k is Boltzman's constant, T is the absolute temperature, and d is the grain diameter.

At higher temperatures the diffusional strain rate is described by the Nabarro-Herring creep equation^[14]:

$$\dot{\epsilon}_s \approx \frac{7\sigma D_v b^3}{kTd^2} \quad \dots[8]$$

where D_v is the volume diffusivity through the grain. From this equation it can be seen that Nabarro-Herring creep is less sensitive to grain size than Coble creep. The major difference between the two is that instead of diffusion along the grain boundaries (Coble creep), vacancies and atoms diffuse through the crystal lattice along a stress gradient (Nabarro-Herring creep) as shown in Figure 13. Again, atom movement happens concurrently in the opposite direction to the vacancy diffusion. Since diffusion becomes

more difficult as the temperature decreases, diffusional creep is limited to temperatures higher than about 40% of the absolute melting temperature (T_m) of the metal. The primary creep regime is not modeled by either Nabarro-Herring or Coble creep^[24].

2.1.4.2 DISLOCATION CREEP

Dislocation creep is a high stress mechanism involving dislocation climb^[14]. When an edge dislocation encounters a dislocation barrier, it can climb around the barrier if there is sufficient energy (i.e. a combination of applied stress and temperature). To move the dislocation a half-plane up requires vacancies to diffuse to the dislocation line^[14]. At low temperatures this is limited by the diffusion of vacancies along the dislocation line, and at high temperatures it is limited by lattice diffusion^[23]. The strain rate of dislocation creep, involving the climb of edge dislocations, is approximated by^[14]:

$$\dot{\epsilon}_s \approx \frac{ADGb}{kT} \left(\frac{\sigma}{G} \right)^5 \quad \dots[9]$$

where A is a material constant, D is the diffusivity, and G is the shear modulus. Note that there is no dependence on grain size for this mechanism. In many creep situations involving engineering alloys, this creep process dominates the elevated temperature response ($T \geq \frac{1}{2} T_m$).

2.1.4.3 GRAIN BOUNDARY SLIDING

The diffusional creep and dislocation creep mechanisms may superimpose in complicated ways^[23]. An example of this is localized shear deformation at grain

boundaries. This is often referred to as grain boundary sliding and appears to encompass two independent deformation modes^[14]. The shear induced displacement of two grains is depicted in Figure 14. The local stress fields cause diffusion of atoms from regions of compression to regions of tension via a diffusional flow process^[14, 25]. The problem in dealing with grain boundary sliding is that it does not represent an independent deformation mechanism^[14]. Therefore grain boundary sliding is needed for diffusion creep and visa versa. These processes are strongly coupled and mutually accommodating; one cannot exist without the other^[14, 25].

2.1.5 CREEP TESTING

Creep tests are generally performed at a single temperature with a uniaxial load^[26]. Tests are most frequently performed in a stress rupture or tensile test machine similar to that shown in Figure 15^[26]. The stress rupture apparatus of Figure 15 is only capable of providing final elongation of the specimen (ϵ_f) and the time to failure (t_f). For strain measurements, an extensometer must be attached to the specimen. Several extensometer configurations are in use and include the type shown in Figure 16. This extensometer is attached via grooves in the sample and the extensometer knife edges. A linear variable differential transducer (LVDT) is attached to measure elongation, from movement of the extensometer, for time dependent deformation via electronic data acquisition.

Stress increment or decrement creep tests require periodically increasing or decreasing the stress level. This can be done by adding or removing weights to reach the desired

stress level. The change in strain rate can then be monitored and analyzed. For a stress increment test an analysis typically consists of plotting the strain rate versus applied stress. Changes in the slope of the plotted values imply a change in the creep mechanism. For instance a change from a small slope in a low stress region to a large slope in a high stress region may indicate a change from diffusional creep to dislocation creep. This is evident from equation 9 for dislocation creep where the strain rate has a greater dependency on stress than the diffusional creep mechanism (equation 7 or 8). For a stress decrement test, the change in strain rate is also monitored to help determine the controlling creep mechanisms. For instance to determine the back stress of a material, several small stress decrement tests can be performed and the incubation period (see Figure 17a^[121]), corresponding to region C of Figure 12, determined. As the decreased stress approaches the back stress the incubation periods become increasingly longer. Therefore, by plotting the cumulative incubation periods versus the cumulative stress reductions (Figure 17b)^[121], the back stress is determined as the asymptotic value of the remaining stress where the incubation period reaches an infinite value^[12].

2.1.5.1 ANELASTIC CREEP TEST

A specific form of a stress decrement test is the anelastic creep test, which also involves reducing the applied stress during testing. To develop a complete understanding of the physical mechanisms of deformation (including those in the primary region), it is necessary to account for the anelastic strain in a quantitative manner. In particular, it is essential to characterize both the magnitude and kinetics of anelastic strain. Further, to

interpret the results in terms of the physical mechanisms of deformation, the processes that control anelastic strain must be identified^[27]. To study anelastic strain, it must be separated from the elastic and plastic strain responses^[21]. This can be accomplished by monitoring the strain after a complete stress reduction.

Anelastic tests in pure FCC metals^[21], stainless steel^[28], and titanium alloys^[29] indicate two distinct stages during anelastic recovery. The two stages are illustrated in Figure 18^[21] for aluminum. The initial stage is one of rapid recovery while the second stage recovers at a slower rate. For steel it has been suggested that the faster recovery is a result of short dislocation segments moving faster. The slower recovery results from the slower movement of longer dislocation segments. For the titanium alloy, it is postulated that the distribution of non-uniform stresses within the heterogeneous substructure of the material drives the anelastic recovery process. Therefore with large remaining strains, the anelastic strain rate is rapid as a result of thermally activated glide. After a large amount of strain has been recovered, the anelastic strain rate decreases as a result of a thermally activated recovery process^[30]. It has been shown^[22] that the primary creep strain is fully recoverable in the diffusion creep regime. In comparison, the magnitude of forward primary creep strain is much larger than the anelastic strain in the dislocation creep regime.

2.2 Near γ -TiAl Intermetallics

A characteristic of intermetallics is that dissimilar atoms will bond with lower energy atoms as opposed to atoms that are similar. The resulting ordered crystal structure has low internal energy and lower entropy. Consequently, in γ -TiAl intermetallics the Ti-Al bonds have lower energy than the Ti-Ti or Al-Al bonds. Without causing localized disorder the minimum diffusion distance is increased, as a result of the Ti-Al bonding, and therefore, the diffusion rate is lowered which improves many high temperature properties^[31]. The highly ordered crystallographic structure however, is also responsible for several disadvantageous properties as outlined in the following sections.

2.2.1 CRYSTAL STRUCTURE OF γ -TiAl

Near γ -TiAl intermetallics consist of two phases, γ -TiAl and α_2 -Ti₃Al as shown in Figure 19^[32]. The crystal structure of the γ phase is ordered face-centered tetragonal, while the α_2 phase is ordered hexagonal closed packed^[33]. Despite the increasing contribution of entropy to free energy with increasing temperature, both γ and α_2 remain ordered to high temperatures. For 48%Al, the disorder temperatures are approximately 1450°C and 1125°C for the γ and α_2 phases respectively^[33]. For stoichiometric γ , the tetragonality ratio is 1.015. This ratio decreases to 1.01 when the composition of γ falls to 48.5 atomic percent aluminum, and increases to 1.03 as the aluminum content increases to 66 atomic percent^[33]. The crystallographic tetragonality causes non-equivalence of the usual $\langle 110 \rangle$ face centred cubic (FCC) slip directions. Therefore,

when compared to FCC structures, dislocation slip is much more difficult in the γ -TiAl crystal structure, leading to an inherent low tensile ductility^[34] but higher stiffness and strength.

2.2.2 NEAR γ -TiAl MICROSTRUCTURES

The central portion of the most recently revised Ti-Al phase diagram is shown in Figure 3^[6]. Four solid phases are indicated in this figure which include: γ -TiAl, α_2 -Ti₃Al, α -Ti (disordered hexagonal close packed), and β -Ti (body centered cubic). For a composition of Ti-48%Al, the transformation from $\alpha+\gamma$ to α occurs at approximately 1375°C($\pm$ 5°C)^[7], often referred to as the α -transus temperature (T_α). As well, both a eutectoid and a peritectic reaction are evident at approximately 1125°C, 39% Al and 1485°C, 46% Al respectively. Given these equilibrium phases and invariant reactions, a range of microstructures can be produced. The microstructural development depends on the heat treatment conditions and gives rise to a wide range of mechanical properties. Figure 20^[11] illustrates four of the important microstructural morphologies, which are:

- a duplex microstructure which is made up of large α_2/γ lamellar grains and small γ grains, typically $\approx 50\ \mu\text{m}$ and $\approx 15\ \mu\text{m}$ respectively (Figure 20a),
- a near gamma (NG) microstructure which consists of a large volume fraction of equiaxed γ grains (Figure 20b),
- a nearly lamellar (NL) microstructure that is characterized by smaller single phase γ grains (which occupy <40% volume fraction) generally located around the periphery of the larger lamellar grains (Figure 20c), and

- a fully lamellar (FL) microstructure consisting of 100% grains of α_2/γ lamellae (Figure 20d).

An important microstructural feature is the crystallographic orientation relationship that exists between the α_2 and γ lamellar plates, which is $(0001)_{\alpha_2} \parallel \{111\}_{\gamma}$ and $\langle 1120 \rangle_{\alpha_2} \parallel \langle 110 \rangle_{\gamma}$ ^[35]. This γ/α_2 relationship implies that when γ forms from the α_2 phase, the γ lamellae will always be oriented parallel to the basal (0001) plane of the α_2 . It also indicates that lamellar formation is more complicated than the typical diffusional eutectoid transformation, exemplified by the formation of pearlite in eutectoid steels. In fact, because of the strong orientation relationship of the α_2 and γ plates, eutectoid transformations rarely occur in near γ -TiAl compositions of engineering interest. In a transmission electron microscope (TEM) study of near γ -TiAl, it was shown that the γ/α_2 lamellar interface is atomically flat and perfectly coherent^[36] except for the presence of ledges^[35, 37].

Heat treatment in the α single phase field, followed by cooling at moderate rates (i.e. air or furnace cooling) results in the formation of γ lamellae along the α basal planes. Thus grains with a lamellar structure of parallel α_2 and γ lathes (Figure 21a & b) will be produced. During cooling, the $\alpha \Rightarrow \alpha + \gamma$ transformation nucleates at grain boundaries with the nuclei coherent with the matrix of one adjacent grain^[7]. The γ lamellae growth into the coherent grain is relatively rapid, but growth into the adjacent grain appears to be dependent on the local total internal energy available for the transformation. If during

heat treatment in the single phase α region, the α grain boundary energy has decreased so much that there is insufficient internal energy available for γ lamellar growth into the incoherent grain, then a relatively planar grain boundary results (Figure 22a^[7]). If the prior heat treatment in the single phase α region generates α grain boundaries with a higher internal energy (for example by reducing the time in the single phase α region so that grain boundaries do not achieve an equilibrium condition), then γ growth can occur in both the coherent and incoherent grains. It has been demonstrated^[38] that incoherent growth of lamellae have less planar interfaces than lamellae resulting from coherent growth. The incoherent lamellar growth results in the formation of interlocked grain boundaries, as shown in Figure 22b. Therefore, the lamellar and grain boundary morphology depends on the heat treatment conditions (time and temperature) in the single phase α region^[7].

The lamellar grain boundary morphology is also influenced by the cooling rate from the α single phase region. A fast cooling rate produces a smoother grain boundary (Figure 21a) than a slowly cooled sample^[12] (Figure 21b). The primary difference between a fast cool and slow cool is that during slow cooling, diffusional growth is more prevalent. This is due to the extended time at elevated temperatures, allowing more diffusion to occur and producing a more interlocked boundary.

The formation and the width of the lamellae is also dependent on the cooling rate. With a cooling rate slower than about 30°C per minute (typical of furnace cooling), the transformations occurring correspond to^[39]:

where L refers to the lamellar morphology. In this transformation, the γ plates nucleate at grain boundaries with the nuclei coherent to the matrix of one adjacent grain^[40]. At this relatively slow cooling rate the density of γ nucleated is low, which produces a fully lamellar microstructure with widely spaced interfaces (FLw), Figure 21b.

Faster cooling rates (air cooled) delay the precipitation of γ to occur at larger undercoolings and may delay lamellar formation to below the α ordering temperature. Hence with a faster cooling rate, the transformation sequence is^[39]:

As a result of the larger undercooling corresponding to this transformation, more γ lamellae will nucleate. This produces a fully lamellar microstructure with more narrowly spaced interfaces (FLn), as shown in Figure 21a.

Heat treatment in the $\alpha + \gamma$ two phase region produces a “duplex” structure consisting of equiaxed single phase γ grains and lamellar grains (Figure 23). During heat treatment in the $\alpha + \gamma$ phase field, α can nucleate and grow in γ with four possible orientations. These are highlighted in Figure 24, corresponding to the basal plane of α parallel to the four $\langle 111 \rangle$ planes of the γ phase. Then, depending on the cooling rate, regions of α

transform into $\alpha_2 + \gamma$ during cooling according to the transformations of equations 10 or 11. Thus, prior γ grains may contain as many as four lamellar orientations. An advantage of heat treating in this two-phase region is that limited grain growth occurs compared to the α single phase region which results in rapid grain growth^[41].

Cyclic heat treatments in the range of about 10°C below the alpha-transus temperature have been found to produce a fine-grained fully lamellar microstructure (FGFL)^[42] (Figure 25). A typical cycle consists of heating to the desired temperature, holding for a short period (typically less than 10 minutes), and then cooling quickly to room temperature^[42]. The total number of cycles per heat treatment ranges between 3 and 12^[42]. The microstructure consists of coarse γ plates with identical crystallographic orientation and bundles of thin γ and α_2 plates^[41]. The transformations resulting in this microstructure are as follows. During heating, the volume fraction of the α phase increases because it is the dominant phase in this two phase region (Figure 3). However, the $\gamma \Rightarrow \alpha$ transformation does not occur in all grains because of the short exposure time. During cooling, new γ plates must nucleate from the α phase in order to increase the volume fraction of γ , as dictated by the phase diagram. This results in bundles of thin lamellae in grains which contain prior α and γ plates. As well, in γ grains containing prior α plates, further lamellae development may occur. During subsequent heat treatment cycles the lamellar grains that were developed in the first cycle, convert to α grains during heating. At the heat treatment temperature the α grains will grow in size at the expense of smaller adjacent grains, and during cooling they will transform to the

lamellar structure. The γ grains which have not been transformed, become potential nucleation sites for new α plates. The overall grain size is small because the heat treatment time per cycle is short^[41]. The enhanced balance of mechanical properties achieved with a reduced lamellar grain size^[42] will be discussed in the section 2.2.4.1.

2.2.3 MECHANICAL PROPERTIES OF NEAR γ -TiAl INTERMETALLICS

The previous section summarized some of the microstructures of engineering importance that may be produced in near γ -TiAl intermetallics. These microstructural changes significantly alter mechanical properties and for this reason, the following section will describe the mechanical properties typical for near γ -TiAl intermetallics. The four mechanical properties that are of most importance for envisaged applications of near γ -TiAl are tensile strength and ductility, fracture toughness, and creep. A summary of these properties is presented in Figure 26^[33] as a function of the microstructures shown in Figure 20. Figure 26 illustrates that as the grain size increases, the strength and ductility (elongation) decrease, while the creep resistance and fracture toughness increase. This figure also indicates that there is an inverse relationship between fracture toughness and ductility and consequently none of the microstructural conditions exhibit an optimum combination of all mechanical properties.

2.2.3.1 TENSILE STRENGTH AND DUCTILITY

The typical room temperature tensile elongation of γ -TiAl intermetallics ranges from 0.3% to 4% while yield and ultimate tensile strength vary from 250-600 MPa and

300-700 MPa. This variation depends upon the composition, processing method, and microstructure^[9] of γ -TiAl intermetallics. The duplex microstructure (Figure 23) is known to have the highest room temperature tensile ductility due to its small grain size^[33]. Comparatively, the large grained fully lamellar condition (Figures 21a and 21b) generally exhibit poor ductility and lower room temperature strength^[9]. As the grain size of the fully lamellar structure is decreased, strength and ductility increase. A microstructure with large lamellar grains has low ductility^[9] because the high yield stress of the lamellar grain is highly anisotropic with respect to the lamellar orientation. That is, the strength is high when the lamellae are parallel or perpendicular to the loading axis, and low for intermediate orientations^[43].

The tensile strength and ductility is also dependent upon temperature. Near γ -TiAl exhibits a ductile-to-brittle transition between 600°C and 820°C, depending on composition and microstructure^[9]. The duplex microstructure has a lower ductile-to-brittle transition temperature (DBTT), while the fully lamellar microstructure has a higher DBTT. A large increase in both twinning and slip system activity results in a rapid increase of ductility above the DBTT^[9]. Finer lamellar interface spacing, which will be discussed more in section 2.2.4.2, results in better tensile strength below the DBTT^[9]. The variations of strength with temperature also depend on microstructure, with greater high-temperature retention in the fully lamellar than the duplex microstructure^[9].

2.2.3.2 FRACTURE TOUGHNESS

Fracture toughness is the ability of a material to resist crack growth and is influenced by several microstructural factors. Near γ -TiAl intermetallics with lamellar grains generally have a higher fracture toughness because of the redundant work required to break individual lamellae in the path of the crack^[44]. As well, it has been shown that finer lamellar spacing increases the crack resistance in fully lamellar structures^[45]. Clearly, grain orientation will play a role in the fracture resistance of lamellar materials. However, no systematic study has been reported in this regard. For the fully lamellar condition, an interlocked grain boundary is more beneficial for fracture resistance than a smooth grain boundary. This is because the interlocked grain boundary leads to a more tortuous intergranular crack path^[45]. Typical room temperature K_{IC} values are 10-16 MPa $\sqrt{m}$ for the duplex microstructure, while the fully lamellar microstructure has an increased toughness between 20-35 MPa $\sqrt{m}$ ^[9]. The relationship between microstructural type and fracture toughness is therefore the inverse of that between microstructure and ductility. The inverse relationship can be reduced by refining the lamellar grain size^[45].

2.2.3.3 CREEP OF NEAR γ -TiAl INTERMETALLICS

The monotonic creep behaviour of a binary near γ -TiAl has been studied at 760°C and 240 MPa, with the creep results reproduced in Figure 27 and summarized in Table II^[12]. From this figure, it is evident that the duplex microstructure has the shortest time to failure and undergoes the largest amount of strain deformation. In comparison, the fully

lamellar microstructure with narrowly spaced lamellae (FLn) has the longest creep life with the smallest amount of strain deformation. The fully lamellar microstructure with widely spaced lamellae (FLw) falls in between the FLn and the duplex in both time to failure and strain deformation. The increased life and reduced minimum strain rate of the FLn microstructure was found to be the result of the reduced lamellar interface spacing. The reduced lamellar spacing is believed to inhibit dislocation motion^[12, 46, 47]. The large amounts of creep strain observed by the FLw, in comparison to the FLn microstructure, was found to be the result of interlocked lamellae along grain boundaries. This interlocking improves resistance to intergranular cracking, allowing greater total creep strain^[12].

Stress increment and decrement tests have also been undertaken and indicate that for a binary near γ -TiAl, dislocation controlled creep occurs^[12, 47]. Stress decrement tests^[47] have also indicated that the internal stress resisting dislocation glide for an applied stress of 240 MPa will be ≈ 190 MPa and ≈ 130 MPa for the FLn and FLw microstructures of Figure 21 respectively. Immediately after a 90% stress reduction from 240 to 24 MPa, the effective stress will therefore be approximately -166 MPa and -106 MPa for the FLn and FLw microstructures respectively. This magnitude of the negative stress implies that the FLn microstructure will have the faster recovery rate.

2.2.4 MICROSTRUCTURAL FEATURES AFFECTING CREEP OF NEAR γ -TiAl INTERMETALLICS

The fully lamellar microstructure exhibits improved secondary and tertiary creep properties compared to the duplex^[9, 12]. As well, it has been established that the minimum strain rate of the fully lamellar condition is markedly lower compared to the duplex microstructure^[12]. The microstructural features that have been associated with the creep properties of near γ -TiAl are presented in the following sections and include: grain size, grain boundary morphology, volume fraction of single phase gamma grains, lamellar spacing, and lamellar orientation.

2.2.4.1 GRAIN SIZE

Studies have shown that for γ -TiAl with a fully lamellar microstructure, the minimum creep strain rate ($\dot{\epsilon}_{\min}$), which is found in the secondary region of Figure 4, is independent of lamellar grain size between 210 and 1200 μm for 760°C and 276 MPa^[11]. According to equation 9, dislocation controlled creep is independent of grain size. This implies that prior to the onset of tertiary creep, grain boundary reaction controlled creep mechanisms have a minimal effect on the creep $\dot{\epsilon}$. In the tertiary region, a fully lamellar microstructure with a small lamellar grain size exhibits more homogeneous deformation. This allows for a greater tertiary creep strain and increased damage tolerance because the interlamellar and intergranular cracks are arrested and become dormant before they can cause fast fracture^[11].

2.2.4.2 LAMELLAR SPACING

The operative creep mechanism for fully lamellar near γ -TiAl microstructures appears to be dislocation creep as discussed in the previous sections. Thus, it is not unreasonable to suggest that the lamellar interface spacing may influence the propensity for dislocation motion. This is supported by the observation of dislocation pileups at lamellar interfaces in creep deformed fully lamellar structures^[11, 48], indicating that the lamellar interfaces act to inhibit dislocation glide. Research has indicated that the improved creep resistance of fully lamellar structures may be the result of the fine α_2/γ lamellar spacing^[48-52]. Two mechanisms have been suggested to account for this improvement. Firstly, interfaces may act to inhibit dislocation glide such that the fine interface spacing reduces the glide distance^[48-52]. This reduced lamellar interface spacing causes a higher internal stress which resists dislocation glide^[47]. Secondly, interface boundaries could act to restrict dislocation motion parallel to the α_2/γ lamellae, causing bowing of segments between interfaces^[49]. Dislocations bowed across lamellae and interacting with interfaces have been observed in creep deformed material^[37, 48, 49, 53]. However, no experimental evidence comparing ostensibly similar microstructures with different interface spacing is available. Due to the lack of grain size effect^[50], the apparent results of interface spacing have been inferred from examination of creep deformed microstructures^[48, 49] or from similar creep properties for samples with significantly different grain sizes and lamellar volume fraction^[51]. Notwithstanding, decreasing lamellar spacing has been observed to improve the creep resistance of near γ -TiAl^[12].

The section on grain size (2.2.4.1) has highlighted that $\dot{\epsilon}_{\min}$ is independent of grain size. Thus, it is not a coincidence that the narrower lamellar interface spacing has been shown to reduce the creep $\dot{\epsilon}$ due to interactions between dislocations and interface boundaries^[12].

2.2.4.3 GRAIN BOUNDARY MORPHOLOGY

The grain boundary morphology of a near γ -TiAl intermetallic has a large effect on creep. The morphology can be controlled via heat treatment and cooling rate as discussed in section 2.2.2. The interlocked grain boundaries help to improve the tertiary creep resistance by reducing the tendency for intergranular cracking^[11-13]. The effect of morphology on the secondary creep region is suggested to be minimal since, as previously outlined, the minimum creep strain rate is controlled by the mobility of dislocations in the lamellae^[12] and not by grain boundary reaction controlled creep mechanisms. The primary creep region has not been studied extensively, therefore it is inconclusive as to whether an interlocked grain boundary will influence primary creep. However, the primary creep strain rate should be influenced by intragranular microstructural features because the minimum creep strain rate is dominated by dislocation glide and climb within the γ lamellae.

2.2.4.4 SINGLE PHASE γ GRAINS

The existence of single phase γ grains in a predominantly lamellar microstructure have an adverse effect on creep. This is a result of the large amount of deformation the γ

grains experience in comparison to lamellar grains^[12]. The effect of these grains along lamellar grain boundaries is two-fold. Firstly, the presence of smaller γ grains will increase the grain boundary area. This allows grain boundary sliding to occur more easily than if no γ grains were present. Secondly, localized dynamic recrystallization of the γ grains has been observed in the duplex structure. This recrystallization effectively reduces the γ grain size and increases the tendency for grain boundary sliding. Both of these factors increase $\dot{\epsilon}_{\min}$ and cause an earlier transition to tertiary creep, shortening the creep life of near γ -TiAl^[11, 12].

2.2.4.5 LAMELLAR ORIENTATION

The lamellar grain orientation with respect to the stress axis is another factor which can affect the properties of a near γ -TiAl material. Grains that are parallel or perpendicular to the stress axis are referred to as being in a 'hard' orientation. This is in contrast to the 'soft' or 'easy' orientation which refer to grains with lamellae having angles between 30° and 70°^[11]. Properties of TiAl lamellar single crystals as a function of lamellar orientation have been examined and are presented in Figure 28^[43]. This figure shows that hard mode deformation causes maximum strength but relatively low ductility. In comparison, soft mode deformation causes low strength while ductility is at a maximum. Soft mode deformation is controlled by shear deformation of the γ phase producing $\gamma/\gamma_{\text{twin}}$ interfaces, which are parallel to the original lamellar direction, and $1/2\langle 110 \rangle$ dislocation slip^[43]. The hard mode deformation is controlled by $1/2\langle 110 \rangle$

dislocation slip and intersection of twins in γ lamellae with α_2 lamellae. The hard mode is stronger because α_2/γ interfaces are an effective barrier to twinning and reduce the mean free path for dislocation motion^[43]. Consequently, minimal change occurs in the dislocation substructure of ‘hard’ mode grains during creep deformation. It is therefore evident that creep deformed substructures in each lamellar grain will vary considerably depending on the lamellar orientation of each grain in polycrystalline, near γ -TiAl fully lamellar microstructures^[13].

2.2.4.6 LAMELLAR INTERFACES

Large primary creep strains have been attributed to the abundance of interfaces in the fully lamellar condition^[54], which may act as sources for dislocations^[53]. However, the motion of these dislocations will be restricted by the same abundant interfaces^[53]. The lamellar interfaces have been observed to have a high density of dislocations^[54]. A recent TEM investigation of Ti-47Al-2Cr-1Nb-1Ta suggests that interface dislocations will be the first type to become active during deformation^[55]. As a result, these interface dislocations may pile up and increase stress concentrations significantly to cause the emission of bowed dislocation segments into the matrix^[37, 53].

From the above discussion, it is evident that the overall creep properties can be improved by a number of methods. These include reducing the volume percent of single phase gamma grains, introducing interlocked grain boundaries, reducing the lamellar spacing, and orientating the lamellar grains in the ‘hard’ mode.

2.3 Summary

The information presented in the preceding sections highlight several important factors regarding near γ -TiAl intermetallics, including:

1. near γ -TiAl intermetallics are emerging as a material of substantial engineering importance,
2. the mechanical properties of near γ -TiAl intermetallics are dependent on microstructure,
3. near γ -TiAl intermetallics exhibit favourable creep properties, with the fully lamellar microstructure being the most creep resistant, and
4. the primary creep behaviour, which is of importance for many gas turbine applications, has not been extensively characterized for near γ -TiAl intermetallics and therefore, the microstructural features and mechanisms controlling the primary creep resistance are not well established.

Useful information concerning the creep behaviour of engineering materials can be determined by studying the anelastic creep response. However, there is no existing literature describing the anelastic creep behaviour of near γ -TiAl intermetallics.

CHAPTER 3

THESIS OBJECTIVE AND SCOPE

In light of the findings of the literature review, the specific objective of this thesis is to determine the primary creep properties of a binary near γ -TiAl intermetallic as a function of microstructure and applied stress. Given the value of analyzing the anelastic strain behaviour to determine the creep mechanisms in other alloy systems, this thesis research includes as an objective, the determination of the anelastic strain response of the near γ -TiAl intermetallic during primary creep.

To achieve the desired objectives, the completion of the following tasks is required:

1. Development of several near γ -TiAl microstructures, including several fully lamellar morphologies and the duplex microstructural condition. These microstructures are developed via controlled heat treatments. The heat treated microstructures are fully characterized.
2. Creep testing at two stress levels to predetermined strain levels within the primary region of the microstructures developed in step 1. The anelastic strain response is determined following near total stress reductions from these

predetermined strain levels. This task includes the development of suitable data acquisition procedures to measure the anelastic time response.

3. Examination of the creep deformed microstructures, via optical and transmission electron microscopy, to help determine the operative deformation mechanisms controlling the primary creep behaviour of near γ -TiAl intermetallics.

The next chapter outlines the materials and experimental procedures followed in completing these tasks. The following chapters present the results and discuss the significance of the findings.

CHAPTER 4

MATERIALS AND EXPERIMENTAL PROCEDURE

4.1 Materials

For this thesis, metal powder produced via the titanium gas atomization (TGA) process (illustrated in Figure 29) was obtained from Crucible Compaction Metals. In the TGA process, a 27 kg charge is melted in a cold wall induction crucible. By utilizing the cold wall, a thin skull of the charge remains in the crucible. This helps to promote cleanliness without the need for a refractory liner which often causes contamination^[56]. A molten stream is poured unto a tundish with a refractory metal nozzle at the bottom which produces a stream of the molten alloy. The stream is atomized and solidified by a high pressure jet of argon gas. The powder is typically screened to -35 mesh (500 μm) and processed through a magnetic separator to remove foreign particles that may have been magnetized during atomization. The chemical composition, in weight percent, of the material used for this thesis is given in Table III. The powder was predominately spherical.

4.2 Material Preparation

The progression from powder to the final creep sample can be seen in Figure 30. To can the powder material, a 304 stainless steel tube with a length of 350 mm, inner diameter of 15.9 mm, and a wall thickness of 0.08 mm was initially prepared. The tube was cleaned in an ultrasonic bath of methanol. A hydraulic tool shown in Figure 31 was used to seal the tube by first crimping one end. This was then followed by tungsten inert gas (TIG) welding to form a canister. To ensure that the canister was properly welded with no leaks, it was evacuated for several hours using the vacuum system shown in Figure 32. This system can evacuate three canisters at once and consists of both a roughing pump and a diffusion pump. With a properly sealed canister, the vacuum system could maintain a pressure (in the canister) of between 1×10^{-5} to 1×10^{-6} mbar.

The canister was filled with powder in the glove chamber (Figure 33), in an atmosphere of argon to prevent contamination of the powder. To ensure that no contaminants entered the glove chamber it was flushed three times with argon. The canisters were filled with the binary titanium aluminide powder to a height of approximately 165 mm ($\approx 32 \times 10^3 \text{ mm}^3$). To prevent the powder from contaminating the vacuum system, glass fibre was inserted at the top of the canister. A rubber plug sealed the canister temporarily during the transfer to the vacuum system. The can was evacuated to approximately 8×10^{-6} mbar pressure and degassed at approximately 550°C for at least 1 hour using the high vacuum system shown in Figure 32. After degassing, while still attached to the vacuum system, the canister was crimped and held using the hydraulic

tool so that the vacuum inside the canister was maintained. Above the crimped end, the canister was cut using a hand held cutoff wheel so that the end of the canister could be TIG welded. All welds were examined under an optical microscope for cracks.

The powder-filled canister was consolidated in a hot isostatic press (HIP) of the type shown schematically in Figure 34. The HIP simultaneously applies heat and isostatic pressure to consolidate the powder. The heating rate of the press is approximately 700°C per hour, and has an inherent power off cooling rate of approximately 750°C per hour. The canisters were placed vertically in an alumina crucible, in the heating chamber of the press, with silica sand surrounding the canisters to ensure they stayed straight during the HIP'ing process. Several thermocouples were attached to the crucible to monitor and control the temperature. The HIP vessel was flushed with argon to remove all air. For this thesis, the HIP conditions were 206 (± 2) MPa at 1250 (± 10)°C for a period of 2 hours. The chamber was pressurized to 55 MPa at room temperature with the remaining pressure generated by the heating cycle. This HIP cycle has been shown to produce >99.9% fully dense samples for γ -TiAl intermetallic powder^[57]. Nevertheless after HIP'ing, the samples were cut and checked for porosity by hand-polishing a small portion of the sample and optically examining it.

The stainless steel canister was removed from the consolidated near γ -TiAl material by machining to approximately 10 mm in diameter. After machining, the bulk material was cut into 80 mm cylindrical rods and cleaned in a methanol bath. The samples were

wrapped in tantalum and encapsulated in quartz tubes that were evacuated and back-filled with argon to approximately 1/3 atm (220 mm Hg). This was done to protect the samples from oxidation during high temperature heat treatment. The tantalum serves as a diffusion barrier between the TiAl and quartz, while the argon pressure prevents the quartz tube, which softens during the high temperature heat treatments, from collapsing.

4.3 Heat Treatment

Several different heat treatments were performed in a high temperature radiation furnace with a type S (platinum/rhodium) reference thermocouple placed beside the sample. The heating rate of the samples was approximately 6°C per second. The heat treatment temperature and soak time varied according to the sample and is summarized in Table IV. All samples were cooled within the quartz tubing. Finally, the samples were subjected to a stabilization heat treatment of 950°C for 48 hours and air cooled.

To examine the heat treated microstructure, a small portion was removed from the end of the heat treated specimen. The sample was polished using an electropolishing technique. This involved mechanically polishing the specimen first to 800 grit, then submersing in a methanol, butanol, perchloric acid solution (64:31:5 by volume), and cooled using liquid nitrogen to -30°C. Using the sample as an anode, 30 volts DC and 0.5-1 A was applied for approximately 3 minutes. To reveal the microstructure, the polished surface was etched in a mixture of distilled water, nitric acid, glycerol, and hydrofluoric acid (12:12:25:1 by volume). This was applied for approximately 10

seconds and rinsed with water to stop the etching process. Since hydrofluoric acid chemically attacks glass (i.e. microscope lenses), a thorough cleaning was done with methanol in an ultrasonic bath prior to optical microscopy.

4.4 Creep Testing Procedure

After ensuring that the proper microstructure was attained, the creep samples were sent to be machined using a low stress grinding technique. The dimensions of the creep test specimen are found in the technical drawing in Figure 35.

Creep tests were performed on a Satec Model D constant load creep frame with a lever arm ratio of 20:1. The basic operation of this test frame is similar to the schematic of Figure 15. All tests were carried out at a temperature of 760°C using a Satec Model TCS 1000 furnace controller and a Satec Model SF-17 furnace. The initial stress applied was either 240 MPa or 172 MPa. Strain was measured using a linear variable differential transducer (LVDT) equipped extensometer attached to the specimen via the notches in the shoulder of the gauge length shown in Figure 35. While the sensitivity of the data acquisition board is capable of distinguishing voltage differences of ± 0.2 mV, the LVDT limited this to ± 0.5 mV over a short sample period and ± 1 mV over the course of the test. The LVDT sensitivity values were determined from the sensitivity graph in Appendix A. This arrangement translates roughly into a strain resolution of 0.0018% over a short sample period or 0.0035% over the course of the test, an acceptable level for this thesis. It should be noted that the resolution of the data acquisition system was increased to the

above values after initial anelastic creep tests. Before every test, the LVDT was recalibrated to provide the most accurate strain reading. A typical calibration graph is shown in Appendix A. Data was collected using a Dell 386 computer using a ADAC 5508SHR high resolution (16 bit) data acquisition board and a Fortran program specifically written for this thesis. The Fortran code is listed in Appendix B. The data acquisition system and program are capable of reading 50 samples per second, however data recordings were made every second during periods of stress changes, and every five minutes at other times.

Two types of creep tests were performed: anelastic and interrupted. Typically anelastic tests involve reduction to a minimum stress to keep the testing system aligned and vibration free^[21]. Although test frames have been developed specifically for anelastic creep testing^[58], anelastic data can be obtained from a stress rupture machine (Figure 15) in conjunction with a LVDT (Figure 16). For stress reduction during anelastic creep tests, the stress was reduced manually by removing 90% of the weight after creep to a predetermined strain level of either 0.2, 0.4, or 0.8%. The weights (approximately 14 kilograms) were manually removed at a steady rate to ensure that undue vibrations were not introduced to the load train. The load removal presents a severe obstacle with respect to vibrations induced in the test frame. Therefore, to avoid erroneous results, only time dependent strain is shown after load removal. This is typically accomplished with a *subjective* separation between the elastic and time dependent strain. The interrupted creep tests were stopped after 0.2 or 0.4% strain.

These samples were cooled under load by turning the furnace off and opening the furnace door so that the sample cooled quickly to avoid further creep deformation.

4.5 Microstructural Analysis

Selected samples, which included heat treated, interrupted, and recovered conditions, were analyzed using a transmission electron microscope (TEM). TEM samples of both the creep deformed conditions, interrupted and recovered, were made from the gauge length of creep specimens. With the plane of the sample normal to the stress axis, discs approximately 0.5 mm thick were cut using an Isomet 11-1180 low speed diamond saw. The samples were thinned to between 80 and 100 μm by hand, using 600 grit paper. A Struers Tenupol-3 TEM electrolytic jet polisher was finally used to make a thin foil from the sample with a thickness transparent to the TEM electron beam (≈ 100 nm). This technique involved an electrolytic polishing solution of methanol, butanol, and perchloric acid (64:31:5 by volume) at -45°C , for approximately 2 minutes. The settings on the Tenupol-3 jet polishing apparatus were: an applied potential of 14 volts, a flow rate of 5.5, a current setting of 0.5 amps, and a photosensitivity of 0.7. The polishing was automatically stopped when a hole in the sample allowed passage of an amount of light sufficient to trigger a photosensitive detector. The thin foil was then washed thoroughly in three baths of methanol and dried immediately with filter paper.

CHAPTER 5

RESULTS

5.1 Initial Microstructures

After the material was HIP'ed, examination by optical microscopy indicated that full consolidation was achieved, with porosity rarely observed. The HIP'ed microstructure, shown in Figure 36, consists of α_2 and equiaxed γ grains with an approximate grain size of 15-20 μm . In this figure, the α_2 phase has a lighter contrast and a lower volume fraction than the γ phase. A complete discussion on the evolution of the HIP'ed microstructure is available in reference 59. The HIP'ed microstructure was the initial microstructure for all heat treatments.

The four heat treated microstructures produced for this thesis, as detailed in Table IV, are shown optically in Figure 37. The duplex, FLn, and FLw have been previously characterized by reference 40 with the important characteristics now summarized. The duplex microstructure (Figure 37a) consists of single phase γ grains (typically smaller than 35 μm) and α_2/γ grains (approximately 60 μm). The large grained (approximately 300 μm) fully lamellar microstructures, Figure 37b & c, contain alternating α_2 and γ lamellae plates. The faster cooled FLn microstructure (Figure 37b) had typical γ lamellae

widths of 50-300 nm, an average interface spacing of ≈ 120 nm, and planar grain boundaries. The FLw microstructure (Figure 37c) produced with a slower cooling rate from the heat treatment temperature had γ lamellae between 200 to 1000 nm in width. This gave an average lamellar interface spacing of 450 nm, and well interlocked lamellae along grain boundaries. Finally the cyclic heat treatment produced a fine grain fully lamellar (FGFL) microstructure (Figure 37d). Previous TEM examination of the FGFL microstructure^[41] indicate that the lamellar grains consist of coarse γ plates (as thick as 2.5 μm) interspersed with bundles of much thinner γ and α_2 plates having an average thickness of 100 nm.

For this thesis, TEM examinations indicated a low dislocation density in the as heat treated conditions (i.e. no prior mechanical testing) and is evident in the micrographs shown in Figure 38. As well, the dislocations between lamellae, in the as heat treated condition, appear to be straight. It should be noted that multiple foils and areas were examined such that all TEM micrographs presented show typical areas.

5.2 Mechanical Tests Results

The creep properties of four microstructures were evaluated in the primary creep region with the mechanical test results summarized in Table V. Most of the results presented indicate the creep strain only, and do not include the elastic component of strain that occurs instantly during loading or unloading. The elastic component is the

same for all four microstructures since it is only dependent on the elastic modulus. This is consistent with previous data representations^[12, 41, 46]. Figure 39, illustrates the effect of eliminating the elastic strain corresponding to loading.

To investigate the influence of the applied creep strain on the anelastic response, 90% stress reductions were applied to the FLn condition at 240 MPa for three different strain levels. The forward and anelastic creep results are graphically shown in Figure 40. After the stress reduction, an immediate elastic contraction occurred followed by anelastic recovery. From the forward creep portion of this graph (i.e. before the 90% stress reduction), the data points for each test coincide with the other two tests indicating that the test procedure provided consistent and reproducible results.

The strain response of the four microstructures for an initial stress of 172 MPa and 240 MPa are presented in Figures 41 and 42 respectively. Consistent with the monotonic creep results in Figure 27, there is an improved creep resistance of the fully lamellar microstructures (FLn, FLw, and FGFL) compared to the duplex structure. Also, the differences between the FLn and FLw microstructures appear to be significant; the FLn condition requires more than double the time to achieve a particular strain level.

During the early stages of creep at 172 MPa, a period of very low strain rate sometimes occurs, as is the case for the FGFL condition of Figure 41. This stage, which

lasted nearly 5 hours for the FGFL condition, was apparent for other microstructural conditions, but for shorter time periods.

5.2.1 ANELASTIC ANALYSIS AND RESULTS

The anelastic results are presented in a slightly modified form from that of Figure 18. The data representation of Figure 18 allows determination of both the driving force and kinetics of the anelastic strain. The driving force is associated with the magnitude of the *remaining* strain and the kinetics of the anelastic strain is related to the anelastic strain rate. However, this analytical technique demands the knowledge of the *final* recovered anelastic strain value which may not always be evident. For example, the strain response for the FLn condition for an initial stress of 240 MPa is shown in Figure 43. The final value of recovered anelastic strain could be taken in the region indicated in Figure 43 corresponding to very low strain rates. The difference in strain between any two points in this region, although very small, will have a dramatic effect on remaining strain thereby eliminating the possibility for unequivocal conclusions. Therefore to limit this uncertainty, the *initial* time-dependent strain value (indicated on Figure 43) was used to determine and plot the driving force for anelastic recovery. These plots are shown in Figures 44 thru 46. This analysis method also provides information about the kinetics and driving force of anelastic strain through the slope and magnitude of recovered strain. Calculating the amount of elastic strain using equation 3 can help pinpoint the initial time-dependent strain value.

The anelastic strain response following stress reduction is presented in Figure 44 and illustrates the effect of the different strain levels for the FLn tests shown in Figure 40. In Figures 45 and 46, the anelastic response for all four microstructures is presented for initial stress levels of 172 MPa (after 0.2% strain) and 240 MPa (after 0.4% strain) shown in Figures 41 and 42 respectively. From the three graphs of strain response (Figures 44, 45 and 46), it is evident that all microstructures exhibit two stages of anelastic recovery which are delineated by a change in the anelastic strain rate. This phenomenon is found in Figure 18 and has been observed in previous anelastic measurements^[21, 22, 30]. The recovery strain rate, relaxation time constant, recovery time, and total recovered strain are summarized in Tables VI and VII for the tests shown in Figure 45 and 46 respectively. In both of these tables, the same trends exist between the different microstructures at the low (Table VI) and high (Table VII) applied stress levels.

5.3 Deformed and Anelastic Recovered Microstructures

A microstructural examination was done of the FLn and FLw microstructures. As mentioned previously, some mechanical tests were interrupted at a particular strain level, while others were anelastically recovered. Both of these test stop points are indicated on Figure 40. The following sections represent the two types of tests performed; deformed microstructures (that have not been recovered), and anelastic recovered microstructures.

5.3.1 DEFORMED MICROSTRUCTURES

Typical deformed microstructures for creep tests interrupted after applied stresses of 172 MPa and 240 MPa are shown in Figures 47 and 48 respectively. From both figures it is evident that there are an increased number of dislocations compared to the as heat-treated condition (Figure 38). In the micrographs, after an applied stress of 172 MPa, there are a large number of dislocation loops beginning and ending at the same lamellae interface. As well, there are some dislocations bowed between lamellae. There appears to be three distinct microstructural differences in the samples deformed at 240 MPa in comparison to those at 172 MPa. Firstly, the frequency of dislocations emanating from the lamellae interfaces is reduced for the samples deformed at 240 MPa. Secondly, there is an increased number of dislocations bowed between the lamellae. Finally, the radius of curvature of the bowed dislocations between lamellae is greater for the samples deformed at 240 MPa (Figure 48) than those deformed at 172 MPa (Figure 47) or in the as heat-treated condition (Figure 49).

5.3.2 ANELASTIC RECOVERED MICROSTRUCTURES

Typical FLn and FLw microstructures after anelastic recovery are shown in Figure 49. In contrast to the deformed microstructures which have not been recovered, there is a reduction in the dislocation density. As well, the radius of curvature of the dislocations bowed between lamellar interfaces has decreased. The dislocation loops emanating from the lamellae interfaces were not observed in the recovered microstructures.

CHAPTER 6

DISCUSSION

As mentioned in section 2.2.4.5, the deformation of lamellar grains depends on the orientation to the stress axis. During creep deformation, ‘hard’ mode grains experience minimal change in the dislocation substructure. In the current work, many lamellar grains exhibit a creep deformed structure virtually unchanged from that of the undeformed state. For this reason, attention was directed to regions showing marked differences from the undeformed condition. Based on the previous conclusions^[13], it is reasonable to suggest that examined grains were not in the ‘hard’ mode orientation.

The difference in grain boundary morphology and lamellar interface spacing between the FLn and FLw conditions is significant as indicated in Table II. As discussed previously in section 2.2.4.3, grain boundary reaction controlled creep mechanisms have a minimal effect on the minimum creep $\dot{\epsilon}$, indicating that interpretation of the creep response should consider intragranular deformation. Since lamellar interface spacing is a significant intragranular microstructural feature, it is reasonable to suggest that this will be a major factor influencing the primary creep regime.

In section 2.2.4.6 the supposition that lamellar interfaces are responsible for large primary creep strains was presented. However as shown in Figures 41 and 42, the fully lamellar condition exhibits a longer time to 0.2% and 0.4% creep compared to the duplex conditions at both low and high applied stresses respectively. From an application viewpoint it is significant that the time to a specific strain level, for example 0.2% and 0.4% in Figures 41 and 42 respectively, is always greater for the fully lamellar (FL) structures. This result is consistent with the suggestion (section 2.2.4.6) that although the FL structure has an abundance of interfaces which may act as dislocation sources, the motion of these dislocations is restricted by the same abundant interfaces. In contrast, the duplex condition contains fewer interfaces, and therefore a potentially lower dislocation density. The dislocations however, can glide over a greater distance^[53] which results in greater primary creep strain. Therefore, in addition to the longest creep life and lowest $\dot{\epsilon}_{\min}$, the fully lamellar condition exhibits the best primary creep behaviour.

The FGFL condition exhibits an improved creep resistance compared to both the duplex and FLw microstructures. A slight reduction in creep resistance of the FGFL with respect to the FLn condition is most likely a result of the coarse γ lamellae found in the FGFL microstructure (section 2.2.2). Given the improved properties of creep life and tensile strength^[42], this thesis further illustrates the potential of the FGFL microstructure with respect to primary creep behaviour.

For the FLn condition, it can be seen from Figure 44 that the anelastic recovery after applied strains of 0.4% and 0.8% are similar and occur with faster kinetics than that of the 0.2% strained sample. This indicates that the strain rate is in a pseudo steady state condition for the 0.4% and 0.8% samples. That is, the primary creep transient requires $<0.4\%$ creep strain but $>0.2\%$ creep strain. This is deduced as a result of saturation of the magnitude of the anelastic strain which occurs as the primary creep strain is exhausted. This phenomena has been observed and suggested previously as discussed in section 2.1.3.

The dislocation loops emanating from the lamellae interfaces after low and high stress are illustrated in Figures 47 and 48 respectively. Similar dislocation structures have been observed previously after deformation^[53, 37]. The observation of these dislocation loops after only 0.2% creep strain, when the dislocation density in the γ lamellae is still very low (Figure 47), suggests that the loops emanate from interfaces without the requirement for interaction between dislocations in the γ lamellae and interfaces. No loops were observed in the microstructures before deformation (Figure 38), suggesting that they may be generated by a combination of the interface coherency stresses and an externally applied creep stress.

As mentioned in section 2.1.2, the dislocation density will increase during the early stages of deformation with one obvious source being the dislocation loops. With continued applied stress, the ends of the dislocations loops appear to expand until

dislocation segments spanning the γ lamellae develop. It is suggested that the interfaces act on these dislocations in a similar fashion to Orowan bowing in particle strengthened alloys. Therefore the glide of the dislocations segments along the lamellae interact with the interface, leading to the bowed morphology of the dislocations observed in Figures 47 and 48. The reason why the interfaces act to hinder the dislocation motion is suggested to be related to the existence of interface ledges (section 2.2.4.6) which would require the motion of such segments to be a combination of both climb and glide. Therefore it is postulated that the reduced number of interfaces in the FLW microstructure reduces the Orowan stress (equation 2) necessary for dislocation motion along interfaces. Consequently, there is greater mobility of dislocations within the γ lamellae which in turn, contributes to faster creep strain rates.

As determined from the above discussion, the primary creep deformation of fully lamellar structures is controlled by the emission of dislocations from the lamellae interfaces. As well, it is also controlled by the mobility of these dislocations along the γ lamellae via combined glide and climb. It is speculated that initially, the rate at which dislocations are emitted from the interfaces is the dominant factor for the low primary creep strains. The ease of mobility of the emitted dislocations becomes increasingly important for larger strains in the primary region.

Since Ti-48Al forward creep at 240 MPa is thought to occur by dislocation glide^[43, 46], it is reasonable to suggest that glide will influence the anelastic response.

The microstructural feature controlling the glide distance will be the γ grain size for the duplex condition and the lamellar interface spacing in the fully lamellar conditions. Since the latter is more than an order of magnitude smaller, the extent of reverse glide during anelastic recovery is also smaller as indicated in Tables VI and VII.

Several dislocation processes are responsible for the anelastic reverse strain following stress reduction. The significantly straighter dislocations spanning the γ lamellae after recovery (Figure 49) compared to the bowed dislocations existing after forward creep at 240 MPa (Figure 48), may indicate that dislocation unbowing causes reverse glide of dislocations spanning γ lamellae. It is expected that this reverse glide occurs rapidly after the stress reduction and accounts for the relatively fast anelastic strain rate found in stage I (Tables VI and VII). The lower dislocation density after anelastic recovery may indicate dislocation annihilation, which has been previously suggested to occur in $\text{Ti}_3\text{Al+Nb}$ ^[22]. The lack of dislocation loops associated with lamellar interfaces after recovery, further suggests that many dislocations are annihilated by being re-absorbed into the interfaces. This could require slow recovery processes which would account for the prolonged period of anelastic strain recovery in stage II.

During anelastic recovery at 172 MPa as well as 240 MPa (Tables VI and VII respectively) the strain rate is faster for the FLn condition compared to the FLw condition. That is, the kinetics of the anelastic recovery is faster for the FLn condition. This may seem unusual since the forward creep resistance of the FLn microstructure is

considerably better due to the narrow interface spacing. From section 2.2.3.3, it was shown that the FLn microstructure has a larger back stress resisting dislocation glide. The magnitude of the back stress immediately after unloading is also larger for the FLn microstructure. This therefore results in a larger driving force for recovery and consequently, faster anelastic kinetics for the FLn condition.

The relaxation time, τ , is longer in the fully lamellar microstructures than the duplex at both applied stresses as indicated in Tables VI and VII. This was expected because an increasing τ indicates a longer time to recover $1/e$ of the final strain value during unloading, which corresponds inversely to a decreasing recovery rate. For the duplex microstructure, both anelastic stages have a significantly higher rate of strain recovery, and thus a smaller relaxation time. However between the fully lamellar microstructures, this inverse trend does not exist because τ increases with the stage I and II strain recovery rates. The reason for this anomaly is unclear at the present time.

CHAPTER 7

SUMMARY AND CONCLUSIONS

- 1) At 240 MPa and 172 MPa and 760°C, the fully lamellar condition exhibits better primary creep properties than the duplex condition.
- 2) Primary creep resistance at 760°C and both 172 MPa and 240 MPa is improved by reducing the lamellar interface spacing.
- 3) The recoverable anelastic strain increases as the applied primary creep strain increases, but remains constant for applied strains corresponding to the *pseudo* steady state regime.
- 4) Dislocation loops emanating from the lamellae interfaces are a potential dislocation generation mechanism for fully lamellar near γ -TiAl microstructures. The dislocations generated may contribute to the primary creep, which appears to be controlled by the combined glide and climb of dislocations within the γ lamellae.
- 5) The anelastic response occurs in two stages. The rapid recovery of the first stage corresponds to the unbowing (or dislocation line tension) of dislocation segments between lamellar interfaces. The kinetically slower second stage corresponds to non-conservative dislocation motion, driven by the internal stress, and dislocation annihilation or re-absorption of dislocations into lamellar interfaces.

7.1 Suggested Future Work

To complement work begun in this thesis, some future work may include investigation of the initial loading characteristics to conclusively determine the reason for the period of very low strain rate, shown in Figure 41. This phenomenon may have been a result of the microstructure. The significance is that if it was a microstructural effect, once the mechanism is determined, it could help improve the creep properties of other materials by reducing the strain rate in the primary region.

An additional future direction would be to examine the current TEM foils at higher magnifications. This would provide useful information in two areas. Firstly, to determine the reason why dislocation loops are being emitted from particular points along an interface. Secondly, to look at the area where bowed dislocations between lamellae interfaces exist and try to determine why they are in fact bowed. Both of these areas would provide important microstructural design information. It is speculated that further research will indicate that the lamellar interface ledges do play an important role in both producing dislocations and restricting the movement of dislocations causing bowing. Therefore a microstructure could be enhanced so that the primary creep rate may be reduced by restricting both the generation and movement of dislocations.

The creep deformed samples of the fine grain fully lamellar (FGFL) microstructure were not examined in this thesis and thus would be another area to investigate. By examining the dislocation substructure of the FGFL condition, further insight to the

mechanisms controlling primary creep may be provided. The finer spacing of some of the lamellae in the FGFL microstructure may reduce the tendency for dislocation generation and increase the stress necessary for dislocation movement.

As mentioned in the discussion of this thesis, the relaxation time constant (τ) produced some anomalies which should be investigated further. The anelastic results may have been too 'noisy' to fit a curve, so it follows that reducing the noise in the data acquisition system should be the first place to start. Introducing a second LVDT to the system would also improve reliability of the data. It is believed that this data analysis technique will provide a consistent way to compare anelastic data.

REFERENCES

1. F.H. Froes, "Advanced Metals for Aerospace and Automotive Use", *Materials Science and Engineering*, **A184**, (1994), p119-133.
2. W.E. Dowling, Jr., J.E. Allison, L.R. Swank, and A.M. Sherman, "Development of TiAl-Based Automotive Engine Valves", *Materials Research Society Symposium Proceedings*, **364**, (1995), p757-767.
3. W.E. Dowling, Jr., J.E. Allison, L.R. Swank, and A.M. Sherman, "TiAl-Based Alloys for Exhaust Valve Applications", *New Engine Design and Engine Component Technology SP-972*, Society of Automotive Engineers, (1993), p31-38.
4. Y-W. Kim, "Technology Transfer", *JOM*, July (1994), p 7.
5. C.M. Austin and T.J. Kelly, "Progress in Implementation of Cast Gamma Titanium Aluminides", *Gamma Titanium Aluminides*, The Minerals, Metals and Materials Society, (1995), p21-32.
6. J.C. Mishurda and J.H. Perepezko, "Phase Equilibria in Ti-Al Alloys", *Microstructure/Property Relationships in Titanium Aluminides and Alloys*, The Minerals, Metals and Materials Society, Warrendale, Pennsylvania, (1991), p3.
7. D. Dudzinski, L. Zhao, J. Beddoes, "Microstructural Evolution of Near- γ TiAl Alloys During Long-Time Heat Treatments", *Scripta Materialia*, **35**, No.3, (1996), p367-372.
8. J.C. Chesnutt, "Titanium Aluminides for Aerospace Applications", *Superalloys 1992*, The Minerals, Metals and Materials Society, (1992), p381-389.
9. Y-W. Kim, "Ordered Intermetallic Alloys, Part III: Gamma Titanium Aluminides", *JOM*, July (1994), p30-39.
10. P. Martin, "Microstructure / Processing of Ductile Gamma Titanium Aluminide Alloys, Interim Report for Period 10 August 1990 through 31 January 1991", *report #WL-TR-91-4056*, Materials Directorate, Wright Laboratory, Wright-Patterson Air Force Base, Ohio, June (1991).
11. J. Beddoes, W. Wallace, and L. Zhao, "Current Understanding of Creep Behaviour of Near γ -Titanium Aluminides", *International Materials Reviews*, **40**, No. 5, (1995), p197-217.

12. J. Triantafillou, The Creep Behaviour of Lamellar Near γ -Titanium Aluminides, M. Eng Thesis, *Carleton University*, (1996), p1-127.
13. W.R. Chen, J. Triantafillou, J. Beddoes, L. Zhao, "Effect of Fully Lamellar Morphology on Creep of a Near γ -TiAl Intermetallic", *Acta Metallurgica Sinica (English Letters)*, **9**, (1996), p565-570.
14. R.W. Hertzberg, Deformation and Fracture Mechanics of Engineering Materials, *John Wiley & Sons*, Toronto, (1989), p57-78, p123, & p145-180.
15. H.W. Hayden, W.G. Moffatt, J. Wulff, The Structure and Properties of Materials, Volume III -- Mechanical Behaviour, *John Wiley & Sons Inc.*, (1965), p51,52,64.
16. J. Goldak, Materials Engineering - 88.270, *Carleton University*, (1970), p67,68.
17. J.P. Hirth, J. Lothe, Theory of Dislocations 2nd Edition, *John Wiley & Sons*, Toronto (1982), p59-94 & p751-762.
18. R.E. Reed-Hill and R. Abbaschian, Physical Metallurgy Principles - Third Edition, *PWS-Kent Publishing Co.*, (1992), p163.
19. F. Garofalo, Fundamentals of Creep and Creep-Rupture in Metals, *The Macmillan Co.*, (1965), p1-83 & p102-200.
20. R.E. Smallman, Modern Physical Metallurgy - 3rd Edition, *Butterworth and Co. Ltd.*, (1970), p204-228.
21. J.C. Gibling and W.D. Nix, "Observations of Anelastic Backflow Following Stress Reductions During Creep of Pure Metals", *Acta Metallurgica*, **29**, (1981), p1769-1784.
22. R.S. Mishra, D. Banerjee, A.K. Mukherjee, "Primary Creep in a Ti-25Al-11Nb Alloy", *Materials Science and Engineering*, **A192/193**, (1995), p756-762.
23. H.J. Frost, and M.F. Ashby, Deformation-Mechanism Maps, *Pergamon Press*, (1982), p1-3, 43-52.
24. G. Malakondaiah, N. Prasad, G. Sundararajan, and P. Rama Rao, "An Analysis of the Transient Stage in Low Stress Viscous Creep" *Acta Metallurgica*, **36**, No. 8, (1988), p2167-2181.
25. J-P. Poirier, Creep of Crystals, High Temperature Deformation Processes in Metals, Ceramics and Minerals, *Cambridge University Press*, (1985), p195.

26. R.W. Evans and B. Wilshire, "Creep of Metals and Alloys", *The Institute of Metals*, (1985), p38-39.
27. J.C. Gibeling, "A phenomenological Description of Creep Transients Based on Anelasticity", *Acta Metall.*, **37**, No. 12, (1989), p3183-3193.
28. W. Beere and I.G. Crossland "Primary and Recoverable Creep in 20/25 Stainless Steel", *Acta Metall.*, **30**, (1982), p1891-1899.
29. R.W. Hayes, "Preliminary Investigation of the Kinetics of Primary Creep of a Two Phase Gamma TiAl Alloy", *Scripta Metallurgica et Materialia*, **29**, (1993), p1229-1233.
30. R.W. Hayes, C. Rhodes, B. London, "Evidence of Heterogeneous Substructure Development During Primary Creep of Ti-6Al-2Sn-4Zr-2Mo", *Scripta Metallurgica et Materialia*, **32**, No. 6, (1995), p867-872.
31. R.L. Fleisher and A.I. Tuab, "Selecting High Temperature Structural Intermetallic Compounds: The Material Science Approach", *JOM*, Sept (1989), p8-11.
32. Y-W. Kim, "Recent Advances in Gamma Titanium Aluminide Alloys", *Materials Research Society Symposium Proceedings*, **213**, (1991), p777-794.
33. Y-W. Kim and D.M. Dimiduk, "Progress in the Understanding of Gamma Titanium Aluminides", *JOM*, **43**, No. 8, August (1991), p40-47.
34. A. Denquin, S. Naka, and T. Khan, "Transformation Kinetics in $\gamma+\alpha_2$ TiAl-Based Alloys", *Proceedings of the 7th World Conference on Titanium*, (1992), p1-7.
35. Blackburn, M.J., "Some Aspects of Phase Transformations in Titanium Alloys", *The Science, Technology, and Application of Titanium*, Pergamon Press, Oxford, 1970, p633-643.
36. G.J. Mahon and J.M. Howe, "Transmission Electron Microscopy Investigation of Interfaces in a Two-Phase TiAl Alloy", *Metallurgical Transactions A*, **21A**, June (1990), p1655-1662.
37. M. Oehring, P.J. Ennis, F. Appel, and R. Wagner, "Microstructural Changes During Long-Term Tension Creep of Two-Phase γ -Titanium Aluminide Alloys", *Materials Research Society Symposium Proceedings*, **460**, (1997), p257-262.
38. A. Denquin, and S. Naka, "Phase Transformation Mechanisms Involved in Two-Phased TiAl-Based Alloys - II. Discontinuous Coarsening and Massive-Type Transformation", *Acta. Mater.*, **44**, No. 1, (1996), p353-365.

39. Y.W. Kim, "Microstructural Evolution and Mechanical Properties of a Forged Gamma Titanium Aluminide Alloy", *Acta Metallurgica Materialia*, **40**, No. 6, (1992), p1121-1134.
40. P.A. McQuay, D.M. Dimiduk, and S.L. Semiatin, "The Decomposition of Alpha Phase During Continuous Cooling and Isothermal Transformation in Gamma Titanium Aluminide", *Scripta Metall. Mater.*, **25**, (1991), p1689-1694.
41. P. Au, A Heat Treatment Procedure to Produce Fine-Grained Lamellar Microstructures in a P/M Titanium Aluminide Alloy, PhD Thesis, *Carleton University*, Ottawa, (1997), p14, 67-69.
42. L. Zhao, P. Au, J. Beddoes, W. Wallace, "Method to Produce Fine-Grained Lamellar Microstructures in Powder Metallurgy, Wrought and Cast Gamma Titanium Aluminides", *US Patent*, #5 653 828, 5 August (1997).
43. H. Inui, M.H. Oh, A. Nakamura, and M. Yamaguchi, "Room Temperature Tensile Deformation of Polysynthetically Twinned (PST) Crystals of TiAl", *Acta Metall. Mater.*, **40**, No.11, (1992), p3095-3104.
44. S. Mitao, S. Tsuyama, K. Minakawa, "Effect of Aluminum Content and Microstructure on Fracture Toughness of Binary TiAl alloys", *Microstructure/Property Relationships in Titanium Alloys and Titanium Aluminides*, The Minerals, Metals and Materials Society, (1991), p213-226.
45. K.S. Chan "Microstructural Effects on Fracture Resistance in TiAl-Based Alloys", *Gamma Titanium Aluminides*, The Minerals, Metals and Materials Society, (1995), p835-847.
46. J. Triantafillou, J. Beddoes, and W. Wallace, "Creep Properties of Lamellar Near γ -Titanium Aluminides", *Canadian Aeronautics and Space Journal*, **42**, No. 2, June (1996), p108-115.
47. J. Beddoes, J. Triantafillou, L. Zhao, "Effect of Stress on Creep of Lamellar Near γ -TiAl", *Materials Research Society Symposium Proceedings*, **460**, (1997), p293-298.
48. B.D. Worth, J.W. Jones, and J.E. Allison, "Creep Deformation in Near- γ TiAl: II. Influence of Carbon on Creep Deformation in Ti-48Al-1V-0.3C", *Metallurgical and Materials Transactions A*, **26A**, November (1995), p2961-2972.
49. J.N. Wang, A.J. Schwartz, T.G. Nieg, C.T. Liu, V.K. Sikka, and D. Clemens, "Creep of a Fine-Grained, Fully-Lamellar, Two-Phase TiAl Alloy at 760°C", *Gamma Titanium Aluminides*, The Minerals, Metals & Materials Society, (1995), p949-958.

50. D.I. Kimm, and J. Wolfenstine, "Effect of Grain Size on the Creep Behavior of Fully Lamellar Transformed γ -TiAl", *Scripta Metallurgica et Materiala*, **30**, No. 5, (1994), p615-619.
51. R.W. Hayes, and P.A. McQuay, "Creep Deformation and Damage Behavior of a Fine Grained Fully Transformed TiAl Alloy", *Microstructure/Property Relationships of Titanium Alloys*, TMS, Warrendale, (1994), p267-277.
52. R.W. Hayes, and B. London, "On the Creep Deformation of a Cast Near Gamma TiAl Alloy Ti-48Al-1Nb", *Acta Metall. Mater.*, **40**, No. 9, (1992), p2167-2175.
53. M.A. Morris and T. Lipe, "Deformation Mechanisms Responsible for the Creep Resistance of Ti-Al Alloys", *Materials Research Society Symposium Proceedings*, **460**, (1997), p275-280.
54. M. ES-Souni, A. Bartels, R. Wagner, "Creep Behaviour of a Fully Transformed Near γ -TiAl Alloy Ti-48Al-2Cr", *Acta Metall. Mater.*, **43**, No. 1, (1995), p153-161.
55. L.M. Hsiung, A.J. Schwartz, and T.G. Nieh, "In Situ Observation of Deformation-Induced Interface Migration in a Fully-Lamellar TiAl Alloy", *Scripta Materialia*, **36**, No. 9, (1997), p1017-1022.
56. C.F. Yolton, and D. Eylon, "Effects of Processing and Heat Treatment on Microstructures and Mechanical Properties of Gamma Titanium Aluminide Powder Compacts", *Titanium '92 Science and Technology*, The Minerals, Metals and Materials Society, (1993), p1083-1090.
57. L.Zhao, J.Beddoes, D. Morphy, W. Wallace, "Microstructure and Mechanical Properties of a PM TiAl-W Alloy Processed by Hot Isostatic Pressing", *Materials Science and Engineering*, **A192/193**, (1995), p957-964.
58. R.S. Mishra and A.K. Mukherjee, "Primary Creep in Aluminum Matrix Composites", *Proceedings of the 7th International Conference on Creep and Fracture of Engineering Materials and Structures*, (1997), p237-246.
59. J.C. Beddoes, W. Wallace and M.C. De Malherbe, "Densification of γ -TiAl Powder by Hot Isostatic Pressing", *International Journal of Powder Metallurgy*, **28**, No. 3, (1992), p313-325.

TABLES

Table I - Comparison of the Mechanical Properties of Near γ -TiAl with Titanium Alloys and Superalloys^[8].

Property	Near γ -TiAl	Titanium Alloys	Superalloys
Density (g/cm ³)	4.04	4.54	8.3
Elastic Modulus (GPa)	176	110	207
Maximum Creep Temperature (°C)	900	540	1090
Maximum Oxidation Temperature (°C)	815	590	1090
Room Temperature Ductility (%)	1-3	15	3-10
Operational Temperature Ductility (%)	5-12	15	10-20

Table II - Summary of Microstructural Features and Monotonic Creep Results at 240 MPa and 760°C^[12].

Property	Microstructure		
	duplex	FLn	FLw
Heat Treatment [*]	1350°C/2h/AC	1380°C/1h/AC	1400°C/½h/FC
Grain Size (µm)	<35(S) 60 (L)	290 (L)	325 (L)
Average Interface Spacing (nm)	N/A	120	450
γ Lathe Width (L, nm)	N/A	50-300	200-1000
Grain Boundary Morphology	planar	planar	interlocked
Creep Life (hours)	68.5	608	279
Minimum $\dot{\epsilon}$ (h ⁻¹)	1.7×10^{-3}	5.9×10^{-5}	2.7×10^{-4}
Fracture Strain (%)	36.4	6.5	18.4

^{*} - All Microstructures were given a stabilization heat treatment of 950°C/48h/AC

h - hour

AC - Air Cooled

FC - Furnace Cooled

S - refers to single phase γ grains

N/A - Not Applicable

L - refers to lamellar grains

Table III - Chemical Composition of Testing Material -- Ti-48Al.

Element	Weight Percent
Aluminum	34.34 (48.1 atomic percent)
Iron	0.1
Carbon	0.011
Nitrogen	0.004
Oxygen	0.053
Titanium	65.492

Table IV - Summary of Heat Treatments.

Microstructure	Heat Treatment		
	Time	Temperature	Cooling Rate
duplex	2 hours	1350°C	6°C/second
FLn	1 hour	1380°C	6°C/second
FLw	½ hour	1400°C	0.3°C/second
FGFL*	10 minutes/cycle	1373°C	8°C/second

* Six cycles were necessary to produce the FGFL microstructure used in this thesis.

Table V - Summary of 760°C Creep Test Results.

Microstructure	Stress (MPa)/ Maximum Strain	Time to Maximum Strain (hours)	Total Anelastic Strain ($\times 10^{-4}$)	Relaxation Time τ [hours]
duplex	172 / 0.2	7.9	9.9	1.91
	172 / 0.2	6.5	3.9	1.54
	240 / 0.4	2.9	7.5	0.43
	240 / 0.4	1.6	--	--
	240 / 0.4	1.1	11.9	0.19
FLn	172 / 0.2	78.6	12.8	51.21
	172 / 0.2	42.8	8.7	6.31
	172 / 0.2	44.4	--	--
	240 / 0.2	6.8	5.0	7.70
	240 / 0.2	5.5	5.5	1.90
	240 / 0.4	14.7	--	--
	240 / 0.4	12.2	9.1	3.36
	240 / 0.4	15.5	9.1	4.31
	240 / 0.4	17.5	8.8	3.09
	240 / 0.8	39.5	10.8	3.65
FLw	172 / 0.2	14.6	8.9	3.61
	172 / 0.2	7.12	--	--
	172 / 0.2	57.7	8.6	64.28
	240 / 0.4	4.0	9.3	2.42
	240 / 0.4	3.5	--	--
FGFL	172 / 0.2	33.0	8.5	6.93
	240 / 0.4	9.9	11.3	5.29

Table VI - Anelastic Strain Results After 0.2% Creep Strain at 760°C and 172 MPa.

Property	Microstructure			
	duplex	FLn	FLw	FGFL
Stage I Recovery Strain Rate ($\times 10^{-8}$) [s^{-1}]	19.37	8.44	4.44	10.83
Stage II Recovery Strain Rate ($\times 10^{-8}$) [s^{-1}]	1.03	0.58	0.57	0.76
Relaxation Time, τ [hours]	1.91	6.31	3.61	6.93
Stage I Recovery Time [seconds]	89	311	372	95
Total Recovered Strain ($\times 10^{-4}$) [mm/mm]	9.9	8.7	8.9	8.5

Table VII - Anelastic Strain Results After 0.4% Creep Strain at 760°C and 240 MPa.

Property	Microstructure			
	duplex	FLn	FLw	FGFL
Stage I Recovery Strain Rate ($\times 10^{-8}$) [s ⁻¹]	94.00	33.67	11.59	40.07
Stage II Recovery Strain Rate ($\times 10^{-8}$) [s ⁻¹]	1.70	1.04	0.92	1.23
Relaxation Time, τ [hours]	0.19	4.31	2.42	5.29
Stage I Recovery Time [seconds]	53	294	320	81
Total Recovered Strain ($\times 10^{-4}$) [mm/mm]	11.9	9.1	9.3	11.3

FIGURES

Figure 1: Manufactured near γ -TiAl exhaust valves developed by Ford Motor Company^[3].

Figure 2: Full set of 5th stage low pressure turbine blades fabricated from Ti-48Al-2Cr-2Nb tested by General Electric in a CF6-80C engine^[5].

Figure 3: Central portion of the Ti-Al phase diagram^[6].

Figure 4: Schematic of creep curve.

Figure 5: Dislocation schematics in a simple cubic structure;
(a) edge dislocation^[14], and (b) screw dislocation^[16].

Figure 6: Micrograph of dislocation pileup on two systems against a grain boundary^[14].

Figure 7: Schematic of Frank-Read dislocation source^[14, 17];

- (a) pinned dislocation segment,
- (b) dislocation segment bowing out of its glide plane,
- (c,d) unstable segment beginning to bend around itself,
- (e) completion of closed loop, and
- (f) original line segment with a new dislocation loop.

Figure 8: Micrograph of a Frank-Read dislocation source^[14, 17].

Figure 9: Schematic representation of double cross-slip mechanism^[14].

Figure 10: Schematic representation of strain response to stress.

Figure 11: Graph defining the relaxation time constant^[15].

Figure 12: Schematic strain transient following a reduction in the applied stress^[21];

- Ⓐ instantaneous elastic contraction,
- Ⓑ time dependent backflow,
- Ⓒ region of zero creep rate,
- Ⓓ constant structure forward creep rate, and
- Ⓔ new steady state.

Figure 13: Schematic of vacancy diffusion at high temperatures (Nabarro-Herring creep)^[14].

Figure 14: Grain boundary sliding (GBS) creates voids (gray area) which are concurrently filled by diffusion creep or conversely diffusion creep causes GBS^[25].

Figure 15: Schematic of a constant load creep frame^[26].

Figure 16: Photograph of extensometer.

Figure 17: a) Schematic plot of incubation periods associated with a stress decrement test^[12], and
b) schematic of cumulative incubation period with cumulative stress reductions^[12].

Figure 18: Anelastic strain rate response for Al^[21].

Figure 19: Atomic arrangements of titanium aluminide intermetallic crystal structures^[32];

(a) γ - TiAl with a face centered tetragonal structure, and

(b) α_2 - Ti_3Al with a hexagonal closed-packed structure.

Figure 20: Micrograph of typical microstructures^[11];
 (a) duplex, (b) near gamma, (c) nearly lamellar, and (d) fully lamellar.

(a) air cooled, FLn

(b) furnace cooled, FLw

Figure 21: Typical heat treated microstructures.

(a) 100 hours

(b) 25 hours

Figure 22: Grain boundary morphology of TiAl after 1375°C heat treatments^[7].

Figure 23: Typical duplex microstructure.

Figure 24: Scanning electron microscope-backscattered electron image indicating four orientations of α in γ .

Figure 25: Typical FGFL microstructure.

Figure 26: Effect of microstructure on grain size (GS) and mechanical properties including: fracture toughness, tensile strength and elongation (El), impact resistance (IR), and creep resistance (CR). Creep resistance was defined as the reciprocal of the secondary creep strain rate^[33].

Figure 27: Monotonic creep test results of Ti-48Al at 760°C and 240 MPa^[12].

Figure 28: Properties of lamellar single crystal as a function of lamellar orientation to the applied load^[43];
(a) yield strength, and (b) tensile elongation.

Figure 29: Schematic representation of the crucible refractory free process for titanium powders.

Figure 30: Photograph showing progression of TiAl powder to the final creep specimen;

- (a) powder material,
- (b) stainless steel tube with bottom crimped and welded,
- (c) evacuated tube ready for hot isostatic processing,
- (d) consolidated can,
- (e) bulk material with stainless steel can machined off,
- (f) encapsulated material with ends of sample wrapped in tantalum, and
- (g) creep test specimen.

Figure 31: Photograph showing hydraulically operated crimping tool.

Figure 32: Photograph of vacuum system showing; (a) roughing pump, (b) diffusion pump, (c) filled tube being evacuated, and (d) vertical furnace for degassing.

Figure 33: Photograph of inert gas chamber used to fill stainless steel tubes with TiAl powder.

Figure 34: Schematic representation of the hot isostatic press.

Figure 35: Technical drawing of the creep specimen.

Figure 36: Photograph of HIP'ed microstructure.

(a) duplex

(b) FLn

(c) FLw

(d) FGFL

Figure 37: Typical as heat-treated microstructures.

Figure 38: TEM micrographs of lamellar regions of duplex, FLn, and FLw in the as heat-treated condition.

Figure 39: Comparison of creep results with and without elastic strain portion corresponding to initial loading.

Figure 40: Response of FLn condition at 760°C and 240 MPa initial stress.

Figure 41: Response of FLn, FLw, duplex, and FGFL conditions at 760°C and 172 MPa initial stress.

Figure 42: Response of FLn, FLw, duplex, and FGFL conditions at 760°C and 240 MPa initial stress.

Figure 43: Strain Response of FLn condition at 760°C and 240 MPa initial stress.

Figure 44: Anelastic response following stress reduction at 760°C and 240 MPa initial stress for FLn condition.

Figure 45: Anelastic response of FLn, FLw, duplex, and FGFL conditions after 0.2% creep strain at 760°C and 172 MPa initial stress.

Figure 46: Anelastic response of FLn, FLw, duplex, and FGFL conditions after 0.4% creep strain at 760°C and 240 MPa initial stress.

Figure 47: Typical TEM micrographs of FLn and FLw microstructures after 0.2% deformation at 172 MPa.

Figure 48: Typical TEM micrographs of FLn and FLw microstructures after 0.4% deformation at 240 MPa.

Figure 49: Typical TEM micrographs of FLn and FLw microstructures after recovery.

APPENDICES

APPENDIX A
LVDT SENSITIVITY GRAPHS

LVDT Sensitivity

- this sensitivity graph was produced by running the data acquisition system overnight with the LVDT set in a calibration fixture
 - over the course of the test there was a drift of ± 0.5 mV for short sample periods and ± 1 mV for the total test period

Calibration of LVDT #2

APPENDIX B

FORTRAN SOURCE CODE FOR DATA AQUISITION

[illegible]

Another unique feature of this program is the remote monitoring capability. This is done using a numeric pager which the computer automatically dialed every half hour to provide an update of the current strain level. This is particularly useful at the low stress levels where the strain rate was slow and was necessary instead of manually monitoring the strain level.

```
C* Program written by: David C. Dudzinski
C* Last modified: 28-Nov-1996 by David C. Dudzinski
```

```

C      -- copy datafile to the A drive as A:\DATA
l or L -- insert a line into data file
n or N -- immediate data acquisition
<      -- decrease the time (minutes) between acquiring data
>      -- increase the time (minutes) between acquiring data
-      -- decrease the time (seconds) between acquiring data
+      -- increase the time (seconds) between acquiring data
p or P -- toggle pager mode (Must be turn on initially)
R      -- reset total time and graph area
ESC or 4 -- quit program

```

C* This program also used:

C* To compile:

C*

```

$INCLUDE:'ADLIBI.FOR'
  INTEGER*2 BUFFER(100), COUNT, CHANNEL, TRIGGER, GAIN, LUN, x
  INTEGER*2 textheight /12/, textwidth /6/, lines, texlength
  INTEGER*2 ACQHR / 0 /, ACQMIN / 0 /, ACQSEC / 0 /
  INTEGER*2 LINE / 0 /, HR, MIN, SEC, Pler, PNchr, PRT, Pmin
  INTEGER*2 PhoneStatus / 0 /, DialerStatus, LVDTRes, ComPort
  INTEGER*2 BEGHR, BEGMIN, BEGSEC, junk, GETCHR, STATUS, RANGE
  INTEGER*4 SECONDS, TIMER, VALS / 0 /, PhoneNumber, Pparam
  CHARACTER*9  HWCONFIG, Plinefeed, PReturn
  CHARACTER*12 DATAFILE, LASTTIME / "N/A" /, NEXTTIME
  CHARACTER*12 LASTLVDT, LASTTEMP, TIMEBETW, POINTS
  CHARACTER*15 actualtime / " " /, Ptime
  CHARACTER*24 label, font1 / "t'tms rmn' h12 w6" /
  CHARACTER*40 PSTRING
  CHARACTER*59 COPYSTRING
  REAL*8  VOLTAGE, LVDT, TEMPERATURE, SLOPE, START, TOTALTIME
  REAL*8  TEMPDATA / 0 /, LVDTDATA / 0 /, PhoneTrig
  REAL*8  TEMPDATA2 / 0 /, TEMPDATA3 / 0 /
  LOGICAL KBHIT
  RECORD / xycoord / xy

DO WHILE (junk .EQ. 0)
  STATUS = 0

  CALL clearscreen ($GCLEARSCREEN)
  PRINT '(A)', ' '
  WRITE (*, '(A, \)') ' Enter Name of data file ? '
  READ (*, '(A12)') DATAFILE

  PRINT '(A)', ' '
  PRINT '(A)', ' '
  WRITE (*, '(A, \)') ' Do you wish to use the PAGER option? '

  STATUS = 0
  DO WHILE (STATUS .EQ. 0)
    IF (KBHIT()) THEN
      I = GETCHR()

      IF ((I .EQ. 89) .OR. (I .EQ. 121)) THEN
        STATUS = 1
        PhoneStatus = 1
        DialerStatus = 1      ! Currently ONLY pager option is
                             ! programmed which send the strain
                             ! level every 1/2 hour to a numeric
                             ! pager

        PRINT '(A)', ' '
        WRITE (*, '(A, A)')
        +      '      NOTE THAT THE PHONE NUMBER THAT',
        +      ' WILL BE DIALED'
        WRITE (*, '(A, A)')
        +      '      IS LOCATED IN THE FILE ',
        +      ' CALLED PHONE.DAT.'
      END IF
    END IF
  END DO
END DO

```

```

        OPEN (UNIT = 3, ACCESS = 'SEQUENTIAL',
+           FILE = 'PHONE.DAT', MODE = 'READ', STATUS = 'OLD')
        READ (3, '(I8 / I3 / F11.4)') PhoneNumber, ComPort,
+           PhoneTrig
        CLOSE (UNIT = 3)
        END IF

        IF ((I .EQ. 78) .OR. (I .EQ. 110)) THEN
            STATUS = 1
        END IF

        END IF
    END DO

    PRINT '(A)', ' '
    PRINT '(A)', ' '
    WRITE (*, '(A, \)')
+   ' This program is capable of reading a high resolution',
+   ' (0 - 10v) or a low'
    WRITE (*, '(A, \)')
+   ' resolution (-10 to +10v), which do you prefer (H/L) ? '

    STATUS = 0
    DO WHILE (STATUS .EQ. 0)
        IF (KBHIT()) THEN
            I = GETCHR()

            IF ((I .EQ. 72) .OR. (I .EQ. 104)) THEN
                STATUS = 1
                LVDTRes = 0
            END IF

            IF ((I .EQ. 76) .OR. (I .EQ. 108)) THEN
                STATUS = 1
                LVDTRes = 1
            END IF

            END IF
        END DO

        PRINT '(A)', ' '
        PRINT '(A)', ' '
        WRITE (*, '(A, \)')
+   ' Please enter the slope of the LVDT calibration line (volt/mm)? '
        READ (*, '(F7.4)') SLOPE

        PRINT '(A)', ' '
        PRINT '(A)', ' '
        WRITE (*, '(A, \)') ' Please enter the LVDT start voltage ? '
        READ (*, '(F7.4)') START

        PRINT '(A)', ' '
        PRINT '(A)', ' '

```

```

WRITE (*, '(A, \)')
+   ' Please enter the estimated duration of test (hours)? '
READ (*, '(I4)') RANGE

```

```

SECONDS = 0
TOTALTIME = 0

```

```

PRINT '(A)', ' '
PRINT '(A)', ' '
DO WHILE (SECONDS .EQ. 0)
  WRITE (*, '(A, \)')
+   ' Enter time (in sec) for acquiring data ? '
  READ (*, '(I5)') SECONDS
END DO

```

```

C-----
C-      Convert acquire time from seconds to 24 hour time      -
C-----

```

```

ACQHR = INT (SECONDS/3600)
SECONDS = SECONDS - ACQHR*3600
ACQMIN = INT (SECONDS/60)
SECONDS = SECONDS - ACQMIN*60
ACQSEC = INT (SECONDS)

```

```

C-----
C-      Convert acquire to text so that it can be printed to screen -
C-----

```

```

WRITE (label, '(I3)') ACQHR
TIMEBETW = label(2:3) // ':'
WRITE (label, '(I3)') ACQMIN
TIMEBETW = TIMEBETW(1:3) // label(2:3) // ':'
WRITE (label, '(I3)') ACQSEC
TIMEBETW = TIMEBETW(1:6) // label(2:3)

```

```

C-----
C-      Confirm that entered values are acceptable      -
C-----

```

```

CALL clearscreen ($GCLEARSCREEN)

```

```

PRINT '(A)', ' '
PRINT '(A)', ' '
PRINT '(A)', ' '
PRINT '(A)', ' '

IF (PhoneStatus .EQ. 1) THEN
+   WRITE (*, '(A, A)') ' The phone option will be ',
    ' used with the following parameters:'
    WRITE (*, '(A, I8)') '           Phone # : ', PhoneNumber
    WRITE (*, '(A, I2)') '           COM Port : ', ComPort
    IF (DialerStatus .EQ. 1) THEN

```



```

        WRITE (*, '(A)') '        Dialer Mode : Numeric Pager'
    END IF
END IF

PRINT '(A)', ' '
WRITE (*, '(A, A12)') '        Data file : ', DATAFILE

IF (LVDTRes .EQ. 1) THEN
    WRITE (*, '(A)') ' LVDT Resolution: LOW (-10 to +10v)'
END IF

IF (LVDTRes .EQ. 0) THEN
    WRITE (*, '(A)') ' LVDT Resolution: HIGH (0 to +10v)'
END IF

WRITE (*, '(A, F7.4, A)') '        LVDT Slope : ', SLOPE,
+      ' mm/volt'
WRITE (*, '(A, F7.4)') '        LVDT Start : ', START
WRITE (*, '(A, I4, A)') '        Duration : ', RANGE,
+      ' hours'

PRINT '(A I3 A I2 A I2 A)', ' Data will be recorded every',
+      ACQHR, ' hours, ', ACQMIN, ' minutes, and ', ACQSEC,
+      ' seconds.'

PRINT '(A)', ' '

HWCONFIG = 'TCSH.DAT'
WRITE (*,*) ' Note: ', HWCONFIG, 'must be in current directory'

PRINT '(A)', ' '
PRINT '(A)', ' '
WRITE (*,*) ' Are these values acceptable (Y/N) ? '

STATUS = 0
DO WHILE (STATUS .EQ. 0)
    IF (KBHIT()) THEN
        I = GETCHR()

        IF ((I .EQ. 89) .OR. (I .EQ. 121)) THEN
            STATUS = 1
            JUNK = 1
        END IF

        IF ((I .EQ. 78) .OR. (I .EQ. 110)) THEN
            STATUS = 1
        END IF

    END IF
END DO
END DO

```

```

C-----
C-      Initialize Data File      -
C-----

```

```

OPEN (UNIT = 4, ACCESS = 'SEQUENTIAL', FILE = DATAFILE,
+     FORM = 'FORMATTED', STATUS = 'NEW')

CALL GETDAT (iyr, imon, iday)
WRITE (4, '(A, I2, 1H-, I2, 1H-, I4)') 'File Started ', imon,
+                                     iday, iyr

WRITE (4, '(A)')
+ '  Top      Middle    Bottom    LVDT      Time      Reading    Accum.'
WRITE (4, '(A)')
+ '  (øC)                                Reading          Number    Time (hr) '
WRITE (4, '(A)')
+ '  -----'

CLOSE (UNIT = 4, STATUS = 'KEEP')

```

```

C-----
C-      Initialize Data Acquisition Boards (via adlib function call)-
C-----

```

```

STATUS = INIT(HWCONFIG)
IF (STATUS .NE. 0) GOTO 200

```

```

C-----
C-      Setup Data Acquisition Time
C-----

```

```

CALL GETTIM (HR, MIN, SEC, junk)

```

```

BEGHR = ACQHR + HR
BEGMIN = ACQMIN + MIN
BEGSEC = ACQSEC + SEC

```

```

C-----
C-      Initialize Modem
C-----

```

```

IF (PhoneStatus .EQ. 1) THEN
  Pmin = MIN + 2          ! Time Between Calls (minutes)
  PRT = 2                 ! Number of characters for return
  PReturn = CHAR(13)      ! Return character
  PLinefeed = CHAR(10)    ! Linefeed character
  Pparam = 10100011       ! set modem for 2400, 8-N-1
  CALL INTCOM (ComPort, Pparam, PIer) ! initialize com port

  PNchr = 3
  PString = 'ATZ'
  CALL SENDMC (ComPort, PNchr, PString, PIer)
  CALL SENDMC (ComPort, PRT, PReturn, PIer)

  PNchr = 11
  PString = 'ATL3 S7=40'
  CALL SENDMC (ComPort, PNchr, PString, PIer)
  CALL SENDMC (ComPort, PRT, PReturn, PIer)

  PNchr = 29
  WRITE (label, '(I8)') PhoneNumber

```

```

Pstring = 'ATDT8' // label(2:8) // '.,.,'
WRITE (label, '(F11.5)') LVDT
Pstring = Pstring(1:15) // label(5:5) // '*'
Pstring = Pstring(1:17) // label(7:11) // '***'
WRITE (label, '(I3)') HR
Pstring = Pstring(1:24) // label(2:3)
WRITE (label, '(I3)') MIN
Pstring = Pstring(1:26) // label(2:3) // '#'

CALL SENDMC (ComPort, PNchr, PString, PIer)
CALL SENDMC (ComPort, PRT, PReturn, PIer)

END IF

```

```

C-----
C-      Setup Graphics                                     -
C-----

```

```

junk = setvideomode ( $VRES16COLOR )
junk = setcolor (9)           !bright blue
CALL moveto (0, 80, xy)
junk = lineto (639,80)

CALL moveto (320, 0, xy)
junk = lineto (320, 80)

junk = rectangle ($GBORDER, 0,0,639,479)

junk = setcolor (12)         !red
CALL moveto (20, 100, xy)
junk = lineto (20 , 460)
junk = lineto (635, 460)

junk = registerfonts ( '*.fon' )
junk = setfont (font1)

```

```

C-----
C-      Setup X-Axis titles                               -
C-----

```

```

CALL moveto (19, 461, xy)
CALL outgtext ("0")
CALL moveto (300, 464, xy)
CALL outgtext ("Time (hours)")
CALL moveto (614, 461, xy)
  WRITE (label, '(I4)') RANGE
CALL outgtext (label(1:4))

```

```

C-----
C-      Setup Y-Axis titles      -
C-----

```

```

CALL moveto (2, 85, xy)
CALL outgtext ("% Engineering Strain")
CALL moveto (7, 455, xy)
CALL outgtext ("0")
CALL moveto (5, 95, xy)
CALL outgtext ("10")

```

```

C-----
C-      Place initial text on screen
C-----

```

```

junk = setcolor (15)      !bright white

lines = 2
textlength = 37

CALL setviewport(5, 53, 5+(textlength*textwidth),
+               53+(textheight*lines))
CALL moveto (2,0,xy)
label = "Filename : " // DATAFILE(1:13)
CALL outgtext (label(1:24))
CALL moveto (0, (1*textheight), xy)

```

```

label = " Created on"
WRITE (actualtime, '(I3)') iday
label = label(1:12) // actualtime(1:3) // '-'
WRITE (actualtime, '(I3)') imon
label = label(1:16) // actualtime
WRITE (actualtime, '(I4)') iyr
label = label(1:19) // '-' // actualtime
CALL outgtext (label(1:24))

```

```

label = " at"
WRITE (actualtime, '(I3)') HR
label = label(1:4) // actualtime (1:3) // ':'
WRITE (actualtime, '(I3)') MIN
label = label(1:8) // actualtime
WRITE (actualtime, '(I3)') SEC
label = label(1:11) // ':' // actualtime
CALL outgtext (label)

```

```

lines = 3
textlength = 17

CALL setviewport(5, 5, 5+(textlength*textwidth),
+               5+(textheight*lines))
CALL moveto (2,0,xy)

```

```

CALL outgtext ("The current time :")
CALL moveto (0, (1*textheight), xy)
CALL outgtext ("Temperature (°C):")
CALL moveto (8, (2*textheight), xy)
CALL outgtext ("LVDT Reading :")

lines = 6
textlength = 19
CALL setviewport(330, 5, 330+(textlength*textwidth),
+               5+(textheight*lines))

CALL moveto (0, (0*textheight), xy)
CALL outgtext ("Recorded at :")
CALL moveto (0, (1*textheight), xy)
CALL outgtext ("Temperature (°C):")
CALL moveto (0, (2*textheight), xy)
CALL outgtext ("LVDT Reading :")
CALL moveto (0, (3*textheight), xy)
CALL outgtext ("Number of Points :")
CALL moveto (0, (4*textheight), xy)
CALL outgtext ("Record Every :")
CALL moveto (0, (5*textheight), xy)
CALL outgtext ("Next Recording :")

```

```

C-----
C-      Body of program that acquires data      -
C-----

```

50 CONTINUE

```

IF (BEGSEC .GT. 59) THEN
    BEGSEC = BEGSEC - 60
    BEGMIN = BEGMIN + 1
END IF

IF (BEGMIN .GT. 59) THEN
    BEGMIN = BEGMIN - 60
    BEGHR = BEGHR + 1
END IF

IF (BEGHR .GT. 23) THEN
    BEGHR = BEGHR - 24
END IF

WRITE (label, '(I3)') BEGHR
NEXTTIME = label(2:3) // ':'
WRITE (label, '(I3)') BEGMIN
NEXTTIME = NEXTTIME(1:3) // label(2:3) // ':'
WRITE (label, '(I3)') BEGSEC
NEXTTIME = NEXTTIME(1:6) // label(2:3)

```

```

C-----
C-      Get a temperature reading from the data acquisition card      -
C-----

```

```

COUNT = 100 ! One Hundred Samples
CHANNEL = 81 ! The T/C is connected to channel 1
TRIGGER = 0  ! Immediate reading
GAIN = 3    ! 0 =  $\pm 12.5$  mV, 1 =  $\pm 25$  mV, 2 =  $\pm 50$  mV, 3 =  $\pm 100$  mV
LUN = 1     ! Current Logical Unit Number of the T/C board

```

```

STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
IF (STATUS .NE. 0) GOTO 250
CALL READTEMP (TEMPDATA, BUFFER, COUNT)

```

```

CHANNEL = 83 ! The TOP T/C is connected to channel 3
STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
IF (STATUS .NE. 0) GOTO 250
CALL READTEMP (TEMPDATA2, BUFFER, COUNT)

```

```

CHANNEL = 85 ! The BOTTOM T/C is connected to channel 5
STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
IF (STATUS .NE. 0) GOTO 250
CALL READTEMP (TEMPDATA3, BUFFER, COUNT)

```

```

C-----
C-      Get a LVDT reading from the data acquisition card      -
C-----

```

```

COUNT = 100      ! One Hundred Samples
CHANNEL = 1        ! The LVDT is connected to channel 1
TRIGGER = 0        ! Immediate reading
GAIN = LVDTRes    ! 0 = unipolar A/D (0-10V), 1 = bipolar A/D ( $\pm 10$  V)
LUN = 2           ! Current Logical Unit Number of the SHR board

```

```

STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
IF (STATUS .NE. 0) GOTO 250

```

```

CALL READVOLTAGE (LVDTDATA, BUFFER, COUNT, GAIN)

```

```

C-----
C-      Get a excitation voltage reading from the data acquisition card-
C-----

```

```

COUNT = 100 ! One Hundred Samples
CHANNEL = 2   ! The Power cable is connected to channel 2
TRIGGER = 0   ! Immediate reading
GAIN = 0      ! 0 = unipolar A/D (0-10V), 1 = bipolar A/D ( $\pm 10$  V)
LUN = 2       ! Current Logical Unit Number of the SHR board

```

```

STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
IF (STATUS .NE. 0) GOTO 250

```

```

CALL READVOLTAGE (VOLTAGE, BUFFER, COUNT, GAIN)

```

```

C-----
C-      Write obtained values to disk      -
C-----

```

```

      OPEN (4, ACCESS = 'APPEND', FILE = DATAFILE)
      WRITE (4, '(F8.1, F8.1, F8.1, F11.5, I4, A1, I2, A1, I2,
+ 3X, I4, F14.7)') TEMPDATA2, TEMPDATA, TEMPDATA3,
+ LVDTDATA, HR, ':', MIN, ':', SEC, VALS, TOTALTIME
      CLOSE (4, STATUS = 'KEEP')

```

```

C-----
C-      Update Screen Data      -
C-----

```

```

      WRITE (LASTTEMP, '(F6.1)') TEMPDATA
      WRITE (LASTLVDT, '(F7.4)') LVDTDATA
      WRITE (POINTS, '(I5)') VALS

```

```

      lines = 6
      textlength = 19          ! Length of permanent corresponding values

```

```

      CALL setviewport( (330 + textlength*textwidth),
+      5, 382+(textlength*textwidth),
+      5+(textheight*lines))
      CALL clearscreen ($GVIEWPORT)

```

```

      CALL moveto (0, (0*textheight), xy)
      CALL outgtext (LASTTIME)           ! Last time recorded
      CALL moveto (0, (1*textheight), xy)
      CALL outgtext (LASTTEMP)           ! Last Temperature reading
      CALL moveto (0, (2*textheight), xy)
      CALL outgtext (LASTLVDT)           ! Last LVDT reading
      CALL moveto (0, (3*textheight), xy)
      CALL outgtext (POINTS)             ! Number of points acquired
      CALL moveto (0, (4*textheight), xy)
      CALL outgtext (TIMEBETW)           ! Time between records
      CALL moveto (0, (5*textheight), xy)
      CALL outgtext (NEXTTIME)           ! Next record time

```

```

C-----
C-      Update Graph -- Graph area: x=616, y=360      -
C-----

```

```

      TIMER = 1

      IF (VALS .NE. 0) THEN
        TIMER = 0
        junk = setcolor (3)

        CALL setviewport(20, 100, 636, 460)
        junk = setpixel ((INT(TOTALTIME*(615/RANGE))),
+      (359-(INT((LVDTDATA-START)*(1/SLOPE)*(100/22.18)*(359/10))))))

        TOTALTIME = TOTALTIME + ACQHR - (ACQMIN/60.0) + (ACQSEC/3600.0)
      END IF

```

```

C-----
C-      Wait for the next time to acquire data      -
C-----

DO WHILE (TIMER .EQ. 0)
  CALL GETTIM (HR, MIN, SEC, junk)
  IF ((BEGHR .EQ. HR) .AND. (BEGMIN .LE. MIN)
+    .AND. (BEGSEC .LE. SEC)) THEN
    TIMER = 1
  END IF

WRITE (label, '(I3)') HR
actualtime = label(2:3) // ':'
WRITE (label, '(I3)') MIN
actualtime = actualtime(1:3) // label(2:3) // ':'
WRITE (label, '(I3)') SEC
actualtime = actualtime(1:6) // label(2:3)

Pparam = MIN
IF ((Pmin .GE. 60) .AND. (MIN .LE. 30)) THEN
  Pparam = Pparam + 60
END IF

IF ((PhoneStatus .EQ. 1) .AND. (Pparam .GE. Pmin)
+   .AND. (DialerStatus .EQ. 1)) THEN

  CALL SENDMC (ComPort, PRT, PReturn, PIer)
  PNchr = 29
  WRITE (label, '(I8)') PhoneNumber
  Pstring = 'ATDT8' // label(2:8) // ',,,'
  WRITE (label, '(F11.5)') LVDT
  Pstring = Pstring(1:15) // label(5:5) // '*' // label(7:10)
  WRITE (label, '(I3)') HR
  Pstring = Pstring(1:21) // '**' // label(2:3)
  WRITE (label, '(I3)') MIN
  Pstring = Pstring(1:25) // label(2:3) // '#'

  CALL SENDMC (ComPort, PNchr, PString, PIer)
  CALL SENDMC (ComPort, PRT, PReturn, PIer)

  Pmin = MIN + 30
END IF

COUNT = 100 ! One Hundred Samples
CHANNEL = 81 ! The T/C is connected to channel 1
TRIGGER = 0 ! Immediate reading
  GAIN = 3 ! 0 =  $\pm 12.5$  mV, 1 =  $\pm 25$  mV, 2 =  $\pm 50$  mV, 3 =  $\pm 100$  mV
  LUN = 1 ! Current Logical Unit Number of the T/C board

STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
  IF (STATUS .NE. 0) GOTO 250

CALL READTEMP (TEMPERATURE, BUFFER, COUNT)

```



```

COUNT = 100      ! One Hundred Samples
CHANNEL = 1       ! The LVDT is connected to channel 1
TRIGGER = 0       ! Immediate reading
GAIN = LVDTRes    ! 0 = unipolar A/D (0-10V), 1 = bipolar A/D (±10 V)
LUN = 2          ! Current Logical Unit Number of the SHR board

STATUS = AIN(BUFFER, COUNT, CHANNEL, TRIGGER, GAIN, LUN)
IF (STATUS .NE. 0) GOTO 250

CALL READVOLTAGE (LVDT, BUFFER, COUNT, GAIN)

lines = 3
textlength = 17      ! Length of permanent corresponding text
CALL setviewport( (5 + (textlength * textwidth)),
+ 5, 46+(textlength*textwidth), 5+(textheight*lines))
CALL clearscreen ($GVIEWPORT)

junk = setcolor (15)
CALL moveto (0,0,xy)
CALL outgtext(actualtime(1:8))

CALL moveto (0,textheight,xy)
WRITE (label, '(F6.1)') TEMPERATURE
CALL outgtext (label(1:6))

CALL moveto (0,(2*textheight),xy)
WRITE (label, '(F7.4)') LVDT
CALL outgtext (label(1:7))

IF (KBHIT()) THEN      !* Check For keyboard
    I = GETCHR()        !* input

    IF (I .EQ. 67) THEN      ! C          (copy file)
        junk = 0

        OPEN (UNIT = 7, ACCESS = 'SEQUENTIAL', FILE = DATAFILE,
+           MODE = 'READ', STATUS = 'OLD')

        OPEN (UNIT = 8, ACCESS = 'SEQUENTIAL', FILE = 'A:DATA',
+           FORM = 'FORMATTED', STATUS = 'NEW', iostat=STATUS)

        IF (STATUS .EQ. 6415) THEN
            OPEN (UNIT = 8, ACCESS = 'APPEND', FILE = 'A:DATA',
+           FORM = 'FORMATTED')
        END IF

        DO WHILE (JUNK .EQ. 0)
            COPYSTRING = ' '
            READ (7, '(A59)', iostat=STATUS) COPYSTRING

            IF (STATUS .EQ. -1) THEN
                junk = 1
            END IF

            WRITE (8, '(A59)') COPYSTRING
        END DO

```

```

        CLOSE (7, STATUS = 'KEEP')
        CLOSE (8, STATUS = 'KEEP')
    END IF

    IF ((I .EQ. 78) .OR. (I .EQ. 110)) THEN      ! n or N
        TIMER = 2                                ! (immediate reading)
    END IF

    IF ((I .EQ. 80) .OR. (I .EQ. 112)) THEN      ! p or P
        PhoneStatus = PhoneStatus + 1            ! (toggle dialer)
        IF (PhoneStatus .EQ. 2) THEN
            Pmin = 0
            PhoneStatus = 0
        END IF
    END IF

    IF ((I .EQ. 52) .OR. (I .EQ. 27)) THEN      ! 4 or ESC
        TIMER = 3                                ! (stop program)
    END IF

    IF ((I .EQ. 76) .OR. (I .EQ. 108)) THEN      ! L or l
        OPEN (4, ACCESS = 'APPEND', FILE = DATAFILE)
        WRITE (4, '(A, I3)')
+      ' ----- ', LINE
        CLOSE (UNIT = 4, STATUS = 'KEEP')
        LINE = LINE + 1
    END IF

    IF (I .EQ. 82) THEN                          ! R (reset graph area)
        CALL setviewport (21, 100, 636, 459)
        CALL clearscreen ($GVIEWPORT)

        OPEN (4, ACCESS = 'APPEND', FILE = DATAFILE)
        WRITE (4, '(A)')
+      ' ----- '
        WRITE (4, '(A, I2, A1, I2, A1, I2, F14.7)')
+      ' Graph area reset at: ', HR, ':', MIN, ':', SEC, TOTALTIME
        WRITE (4, '(A)')
+      ' ----- '
        CLOSE (UNIT = 4, STATUS = 'KEEP')

        TOTALTIME = 0
        TIMER = 1
    END IF

    IF (I .EQ. 43) THEN
        ACQSEC = ACQSEC + 1
        junk = 4
    END IF

```

```

IF (I .EQ. 45) THEN
    ACQSEC = ACQSEC - 1
    junk = 4
END IF

IF (I .EQ. 62) THEN
    ACQMIN = ACQMIN + 1
    junk = 4
END IF

IF (I .EQ. 60) THEN
    ACQMIN = ACQMIN - 1
    junk = 4
END IF

IF (ACQSEC .LT. 0) THEN
    ACQSEC = 59
    ACQMIN = ACQMIN - 1
END IF

IF ((ACQMIN .LT. 0) .AND. (ACQHR .GT. 0)) THEN
    ACQMIN = 59
    ACQHR = ACQHR - 1
END IF

IF (ACQMIN .LT. 0) THEN
    ACQMIN = 0
END IF

IF (ACQHR .LT. 0) THEN
    ACQHR = 0
END IF

IF (ACQSEC .GT. 59) THEN
    ACQSEC = ACQSEC - 60
    ACQMIN = ACQMIN + 1
END IF

IF (ACQMIN .GT. 59) THEN
    ACQMIN = ACQMIN - 60
    ACQHR = ACQHR + 1
END IF

+ IF ((ACQSEC .LE. 0) .AND. (ACQMIN .LE. 0) .AND.
    (ACQHR .LE. 0)) THEN
    ACQHR = 0
    ACQMIN = 0
    ACQSEC = 1
END IF

IF (junk .EQ. 4) THEN
    LASTTIME = TIMEBETW

    WRITE (label, '(I3)') ACQHR
    TIMEBETW = label(2:3) // ':'
    WRITE (label, '(I3)') ACQMIN

```

```

TIMEBETW = TIMEBETW(1:3) // label(2:3) // ':'
WRITE (label, '(I3)') ACQSEC
TIMEBETW = TIMEBETW(1:6) // label(2:3) // '*'

lines = 6
textlength = 19      ! Length of permanent corresponding text

CALL setviewport( (330 + textlength*textwidth),
+               5, 382+(textlength*textwidth),
+               5+(textheight*lines))

junk = setcolor (0)
CALL moveto (0, (4*textheight), xy)
CALL outgtext (LASTTIME)                ! Erase old period

junk = SETCOLOR (15)
CALL moveto (0, (4*textheight), xy)
CALL outgtext (TIMEBETW)                ! New time period

END IF

END IF
END DO

IF (TIMER .EQ. 3) GOTO 280

C-----
C-      Adjust the total time parameter      -
C-----

IF (TIMER .EQ. 2) THEN
  IF (BEGSEC .LT. SEC) THEN
    BEGSEC = BEGSEC + 60
    BEGMIN = BEGMIN - 1
  END IF

  IF (BEGMIN .LT. MIN) THEN
    BEGMIN = BEGMIN + 60
    BEGHR = BEGHR - 1
  END IF

  IF (BEGHR .LT. HR) THEN
    BEGHR = BEGHR + 23
  END IF

  TOTALTIME = TOTALTIME - (BEGHR-HR) - ((BEGMIN-MIN)/60.0)
+             - ((BEGSEC-SEC)/3600.0)
END IF

LASTTIME = NEXTTIME
TIMEBETW = TIMEBETW(1:8)
VALS = VALS + 1
BEGHR = HR + ACQHR
BEGMIN = MIN + ACQMIN
BEGSEC = SEC + ACQSEC

GOTO 50

```

```

C-----
C-----
C--      End of main body (which acquires data)      --
C-----
C-----

```

```

200 CONTINUE
    PRINT '(A I6)', ' INIT error :', STATUS
    GOTO 300

250 CONTINUE
    PRINT '(A I6)', ' AIN error :', STATUS
    GOTO 300

275 CONTINUE
    PRINT '(A)', ' The temperature is out of range!'

280 CONTINUE
    PRINT '(/ / / / /)'

300 CONTINUE

    DO WHILE (x .NE. 1)
        IF (KBHIT()) THEN
            x = 1
        END IF
    END DO

    junk = setvideomode ($DEFAULTMODE)
    CALL unregisterfonts ()
    PRINT '()'
    PRINT '()'
    STATUS = RESTORE()
    RETURN

END

```

```

C -----
SUBROUTINE READTEMP (TEMP, VAR, LENGTH)

INTEGER*2 VAR(100), LENGTH
REAL*8     TEMP, COEFF(0:9), VOLTAGE

    TEMP = 0

DO 1000 x = 1,LENGTH,1
1000    TEMP = VAR(x) + TEMP

VOLTAGE = TEMP/(LENGTH) * .2 / 4096

DATA COEFF                                ! Coefficients for TYPE K T/C
+      /0.226584602D0,
+      24152.10900D0,
+      67233.42480D0,
+      2210340.682D0,
+      -860963914.9D0,
+      4.83506D10,
+      -1.18452D12,

```

```

+          1.38690D13,
+          -6.33708D13,
+          0.0D0/

TEMP = VOLTAGE * COEFF(9)

DO 1100 x = 8,1,-1          ! From 9 to 1 in steps of -1
    TEMP = VOLTAGE * (COEFF(x) + TEMP)
1100 CONTINUE

TEMP = COEFF(0) + TEMP
END

C -----
SUBROUTINE READVOLTAGE (VOLT, VAR, LENGTH, GN)

INTEGER*2    VAR(100), LENGTH, GN
REAL*8       VOLT
REAL*4       CONSTANT

CONSTANT = 2**15 / 10

DO 2000 x = 1,LENGTH,1
    IF ((GN .EQ. 0) .AND. (VAR(x) .GT. 0)) THEN
        VOLT = ((VAR(x) / CONSTANT) / 2) + VOLT
    END IF

    IF ((GN .EQ. 0) .AND. (VAR(x) .LT. 0)) THEN
        VOLT = (10 + ((VAR(x) / CONSTANT) / 2)) + VOLT
    END IF

    IF ((GN .EQ. 0) .AND. (VAR(x) .EQ. 0)) THEN
        VOLT = 5 + VOLT
    END IF

    IF (GN .EQ. 1) THEN
        VOLT = (VAR(x) / CONSTANT) + VOLT
    END IF

2000 CONTINUE
VOLT = VOLT/LENGTH
END
C -----

```

IMAGE EVALUATION TEST TARGET (QA-3)

APPLIED IMAGE, Inc
1653 East Main Street
Rochester, NY 14609 USA
Phone: 716/482-0300
Fax: 716/288-5989

© 1993, Applied Image, Inc., All Rights Reserved