MECHANISMS AND EFFECT OF MICROSTRUCTURE ON HIGH TEMPERATURE DEFORMATION OF GAMMA-TiAl BASED ALLOYS

DISSERTATION

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ABSTRACT

There has been a heightened interest in the high temperature behavior of gamma-based
titanium aluminide alloys in the past decade or so. Despite the large body of work done in
the area of high-temperature creep, the present understanding of creep mechanisms and
the effect of alloying and microstructure is limited. The first part of this investigation
concentrates on the creep behavior in the equiaxed microstructure of γ-TiAl alloys. The
aim is to understand the mechanisms and develop a physically-based model for creep in
the gamma phase. A modification of the classic jogged-screw model has been previously
adopted to explain observations of 1/2[110]-type jogged-screw dislocations in equiaxed
Ti-48Al under creep conditions. The goal of this study is to verify and validate the
parameters and functional dependencies that have been assumed in that model. The
original solution has been reformulated with the aid of analytical modeling, numerical
simulations and Transmission Electron Microscopy. Combining the parameters and
dependencies, derived both from experiment and theory, leads to an excellent prediction
of creep rates and stress exponents. In the second part of this study creep behavior of the
fully lamellar TiAl alloys is investigated. Dislocation structures similar to those observed
in the equiaxed alloys suggests that the jogged-screw model can be adapted for lamellar
alloys. The aim of the model is to predict the unique creep phenomenology of fully
lamellar alloys. The strengths and shortcomings of the model are discussed. Probable low
stress creep mechanisms are also suggested. The origin of fully lamellar alloys' superior creep properties stems from the constrained nature of deformation in the lamellae. The results from stress drop experiments are analysed to explore the origin of the large back stresses associated with the fully lamellar alloys. Reduction of the lamellar spacing is proposed as the best way to lower creep rates. In the final part of this study, the microstructural stability of lamellar alloys during exposure to creep conditions is investigated. A detailed investigation of the creep behavior of the aged (stabilized) and unaged (unstabilized) alloys was carried out and subsequent TEM studies were done to characterize the microstructural changes during creep. Continued loss of metastable $\alpha_2$ is proposed as the reason for the larger strain rates in all stages of creep for the alloy in the unaged condition. Dynamic recrystallization and the formation of equiaxed gamma grains are discussed. These results suggest that microstructural stability is critically important in order to achieve the highest possible creep strengths. The effect of microalloying and the role of precipitation hardening in creep are investigated. The probable mechanisms of primary, secondary and tertiary creep are discussed and methods for improving the creep properties are suggested.
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TABLE OF CONTENTS

Abstract ........................................................................................................... ii
Acknowledgments .......................................................................................... iv
Vita ..................................................................................................................... vii
List of Figures ................................................................................................. xii
List of Tables ................................................................................................... xiv

Chapters:
1. Introduction .................................................................................................. 1
   1.1. Motivation ................................................................................................. 1
   1.2. Microstructural Characteristics ............................................................... 2
   1.3. Effect of Chemistry ................................................................................... 7
   1.4. Creep Deformation in metals .................................................................. 10
       1.4.1. Strain rate dependence on temperature and stress ..................... 11
       1.4.2. Creep Mechanisms ........................................................................ 13
   1.5. Deformation in Gamma TiAl ................................................................. 17
       1.5.1. Common deformation modes in γ-TiAl ........................................ 17
       1.5.2. Deformation of lamellar two-phase alloys ................................. 20
       1.5.3. Creep Deformation ......................................................................... 23
           1.5.3.1. Creep mechanisms .............................................................. 27
           1.5.3.2. Microstructural factors affecting creep in FL alloys ............ 29
   1.6. Modeling of Creep in γ-TiAl alloys ...................................................... 32
       1.6.1. The "rule of mixtures" model ......................................................... 32
       1.6.2. A modified Kelly and Street (KS) composite strengthening model 32
       1.6.3. Micromechanisical Modeling ......................................................... 34
       1.6.4. The modified jogged screw model ............................................. 35
       1.6.5. "Interacting elements" model ....................................................... 36
   1.7. Motivation and objectives of this research .......................................... 37

References .................................................................................................... 39

2. Materials and Experimental procedures ................................................... 53
   2.1. Materials Studied ................................................................................... 53
   2.2. Creep Testing ........................................................................................ 55
   2.3. Transmission Electron Microscopy ....................................................... 59

References .................................................................................................... 61

3. Creep in Equiaxed γ-TiAl ........................................................................ 65
   3.1. Phenomenology of creep in equiaxed microstructures ...................... 65
   3.2. Deformation microstructure ................................................................. 67
   3.3. Formation of tall jogs .......................................................................... 69
APPENDICES
Appendix A. Calibration Curves..........................................................242
Appendix B. Extending the model to $\alpha$-Ti........................................243
Appendix C. Isostrain Averaging........................................................245
LIST OF FIGURES

Figure 1.1. A comparison of Young’s modulus values for various high-temperature alloys [2]…………………………………………………………………………………………………… 45

Figure 1.2. The binary Ti-Al phase diagram [2]…………………………………… 46

Figure 1.3. The crystal structure of γ. The darker atoms are Ti atoms and the lighter ones are Al atoms……………………………………………………………………………… 46

Figure 1.4. The crystal structure of α2. The darker atoms are Ti atoms and the lighter ones are Al atoms……………………………………………………………………………… 47

Figure 1.5 The major types of microstructures for the γ - TiAl based alloys: equiaxed, duplex and fully lamellar [2]……………………………………………………….. 47

Figure 1.6. Atom configuration of {110} planes showing the three types of interfaces in L1₀ structure. (Small circles represent the atom positions on the plane one above or below the plane with large circles. Solid and open symbols refer to Ti and Al atoms……………………………………………………………………………… 48

Figure 1.7. A typical creep curve…………………………………………………… 48

Figure 1.8. The ordinary and superdislocations are indicated for a L1₀ structure…. 49

Figure 1.9. Normalized yield stress and work hardening rate at 1% plastic strain in Ti-51Al-2Mn polycrystals as a function of temperature (in °C). The yield stress anomaly is evident [59]……………………………………………………………………………… 49

Figure 1.10. The orientations of PST crystals characterized by the angle φ subtended by the lamellar interfaces to the loading axis…………………………….. 50

Figure 1.11. Anisotropy of room temperature yield strength and ductility in PST crystals is evident. [2]……………………………………………………………………………… 50

Figure 1.12. A comparison of the creep behavior of single phase gamma, duplex and FL microstructures. The performance of FL alloys is superior to the other microstructures [2]……………………………………………………………………………… 51
Figure 1.13 The minimum strain rate versus stress data for several alloys in the equiaxed microstructure is compared. The stress exponent is between 5 and 6 for all the alloys and for a range of temperatures.

Figure 1.14. The minimum strain rate versus stress data for several alloys in the fully lamellar is compared. The stress exponent is 2-3 at low stresses and is 8-10 at high stresses. The data is for one temperature. The strengthening effect of Cr and Nb at high stresses is evident.

Figure 2.1. Optical micrograph of the Ti-48Al (EQ) alloy.

Figure 2.2. SEM micrograph of the as-solutionized K5 FL alloy.

Figure 2.3. Dimensions of the compression testing sample.

Figure 2.3. The lever arm creep testing frame.

Figure 3.1. The creep curves for the equixed microstructure at different stress levels. The creep transient is “normal”.

Figure 3.2. The same curves as in Figure 3.1, replotted on a log-log scale. The power-law time exponent is between 0.3 and 0.5.

Figure 3.3. Several creep tests conducted under the same creep conditions. The creep behavior for these tests is virtually identical.

Figure 3.4. The minimum creep rate is plotted as a function of stress for two different temperatures. The stress exponent is between 5 and 6.

Figure 3.5. Creep tests conducted at different temperatures at 300 MPa.

Figure 3.6. The minimum creep rate plotted as a function of 1/T for different strain levels.

Figure 3.7. The drop in Young’s Modulus as a function of temperature.

Figure 3.8. The value of the “true” creep activation energy plotted as a function of strain and compared to activation energy for lattice self diffusion $Q_d$ (295kJ/mol) and the activation energy for pipe/grain boundary diffusion (from ref [12]).

Figure 3.9 TEM micrograph showing microstructure after creep at 1041 K and 207 MPa. The presence of cusped screw dislocations is evident.

Figure 3.10 Dark field TEM micrograph ($g = 20\bar{2}$) showing microstructure after...
creep at 1033K and 300 MPa. Numerous cusped screw dislocations and loops are seen.

Figure 3.11 TEM micrograph showing microstructure after creep at 1033K and 300 MPa. Jogged-screw dislocations and dislocation debris observed (g = 202)....

Figure 3.12 Cusped jogged-screw dislocation structures observed by a) Ott [15], and b) Morris et al. [14].

Figure 3.13 Cusped jogged-screw dislocation structures observed by a) Appel et al [16], and b) Sriram et al. [13].

Figure 3.14. Schematic showing the formation of a jog by a double cross-slip process.

Figure 3.15. Jogs are not formed when the kink expansion rate is much larger than the kink nucleation rate.

Figure 3.16. The interaction of two kinks lying on different {111} planes can give rise to the formation of unit jogs. This requires that the kink nucleation rate be comparable to the kink expansion rate.

Figure 3.17. Jog-jog interaction will not give rise to the formation of tall jogs if there is an equal number of positive and negative unit jogs.

Figure 3.18. A possible scenario for the formation of tall jogs. The dislocation can cross-slip in both the forward and backward directions in an attempt to avoid the obstacle (adapted from ref [32]).

Figure 3.19. The advance of a self-similarly moving jogged screw dislocation by a distance a. The force balance at steady state is indicated.

Figure 3.20. The vacancy concentration behind the jog is the superposition of vacancy concentration due to a linear string of n point sources.

Figure 3.21. The concentration profiles around the jog under the CMP and FLML assumptions. The scale of the x and y axis is in n, number of Burger's vectors.

Figure 3.22. The actual value of super-saturation at point O in Figure 3.20, as a function of jog height under the CMP and FLML assumptions.

Figure 3.23. Orders of magnitude change in the jog velocity affects the correcting function only marginally, indicating the insensitivity of this function to jog velocity.
Figure 3.24. Insensitivity of the correcting function to lattice diffusivity............. 122

Figure 3.25. Insensitivity of the correcting function to $v/D_v$ ratio ...................... 122

Figure 3.26. The supersaturation limit is seen to change very little with orders of magnitude change in the $v/D_v$ ratio .............................................................. 123

Figure 3.27. The variation of dislocation density as a function of the resolved shear stress confirms Taylor’s relation ................................................................. 123

Figure 3.28. “Unzipping” process which removes the jogs by purely conservative lateral motion [5] ............................................................... 124

Figure 3.29. A schematic of the jog spacing coarsening process due to the conservative motion of jogs driven by line tension. The X’s denote positive jogs and O’s denote negative jogs. Jog coalescence, annihilation and jog-pair nucleation events are indicated .................................................. 124

Figure 3.30. A schematic of the dynamic simulation of jogged-dislocation motion .. 125

Figure 3.31. The result from a part of one simulation is shown. Evolution of the jogged-screw dislocation and jog spacing coarsening are observed. An overall reduction in the total number of jogs (accompanied by an increase in jog height) is also noted. This simulation was run at 200 MPa, 815 C, with an initial configuration consisting of 50 randomly-signed unit jogs initially separated 20 +/- 10 nm. The scale is in meters ............................................................... 126

Figure 3.32. Jog spacing evolution with time is indicated for different stress levels. The “steady-state” jog spacing is inversely proportional to the applied stress. Jog spacing coarsens rapidly ............................................................ 127

Figure 3.33. The evolution of jog height with time is indicated ....................... 127

Figure 3.34. The simulations predict that the jog spacing is inversely proportional to the applied stress ............................................................... 128

Figure 3.35. Jog spacing distribution at different stress levels derived from actual TEM measurements ............................................................... 128

Figure 3.36. The average jog spacing is measured to vary inversely with stress as predicted by the simulations ............................................................... 129

Figure 3.37 There are three distinct scenarios possible as a function of jog height. Jog dragging at short jog heights, dipole dragging at intermediate jog heights and
dipole bypass at large jog heights...........................................................................

Figure 3.38. TEM micrographs with jogs in jog dragging, dipole dragging and
dipole bypass configuration (single-ended source). Also seen is the formation of
prismatic loops that may form behind dipoles. Sample crept to 1% at 300 MPa and
at 760 °C........................................................................................................... 130

Figure 3.39a. The minimum stress required for each of the processes at different
jog velocities....................................................................................................... 130

Figure 3.39b. The minimum stress required for each of the processes at different
temperatures..................................................................................................... 131

Figure 3.40. The modified critical jog height is compared to the one used by
Viswanathan et al. [8], as a function of stress at 1041 K. Actual measurements of
jog heights indicated by the hollow circles....................................................... 131

Figure 3.41. The modified critical jog height is compared to the one used by
Viswanathan et al. [8], as a function of stress at 1088 K............................. 132

Figure 3.42. Model predictions at 1088K compared with experimental values...... 132

Figure 3.43. Model predictions at 1041 K compared with experimental values...... 133

Figure 4.1. Typical creep curves for the binary Ti-48Al (FL) alloy with two
different lamellar spacings.............................................................................. 165

Figure 4.2. Creep curve of the K5 alloy at 760 °C and 315 MPa....................... 165

Figure 4.3. Consistency of creep results between different samples of the K5 alloy
tested under identical conditions................................................................. 166

Figure 4.4. The creep curve for the binary alloy at two different temperatures..... 166

Figure 4.5. The minimum strain rate as a function of temperature compared for
different alloys and a range of stresses......................................................... 167

Figure 4.6. The minimum strain rate as a function of stress of the various FL alloys
and the equiaxed alloy. Lower creep rates and the existence of two stress regimes
are characteristics of the FL microstructure................................................... 167

Figure 4.7. Subgrain formation seen only at large strains and is restricted to the
widest laths. K5 alloy crept at 760°C, 315 MPa to 4.7 %............................ 168

Figure 4.8 Inhomogeneous deformation microstructure, with smaller dislocation
xvi
densities in the thinner laths and significantly larger densities in the thicker laths. Binary lamellar alloy crept at 768C and 207 MPa…………………………………………………………. 169

Figure 4.9. The presence of hard mode jogged screw dislocations in the binary lamellar alloy crept at 768C and 207 MPa is noted…………………………………………………………. 169

Figure 4.10. The presence of trans-lamellar soft-mode dislocations that are jogged (binary lamellar alloy crept at 768C and 207 MPa)…………………………………………………………. 169

Figure 4.11. BF image (g=(111)) of K5SC unaged sample crept at 315MPa, 815C, 0.41% strain (400s). Most dislocations are hard mode ordinary dislocations. Dislocation activity much higher in the wider laths. Several dislocations are jogged…………………………………………………………………………………………….. 170

Figure 4.12. The jogged dislocation in crept K5 alloy, imaged with a) g = (111) and b) g = (002). Invisibility in case b indicates that the dislocation is an ordinary dislocation. Also note the prismatic loop trailed behind the jog…………………………………………………………………………………………………………………….. 170

Figure 4.13. Different beam directions used to image the [110](111) jogged screw dislocation, a) B = [110], b) B = [201] and c) [311]. Case b and c are oriented at ~40° and ~58° to the glide plane along the screw direction. The increase in separation between the segments attached to the jog with increase in tilting proves that this is a tall jog. A rough estimate of the jog height is 57 nm………………….. 171

Figure 4.14. The same two channeling dislocations viewed along two different beam directions. a) B = [110], g =(111) and b) B = [211], g =(111). Tilting the sample parallel to the lamellar interface shows that channeling dislocations lays out dislocation segments along the interface, as indicated…………………………. 171

Figure 4.15. A schematic of the jog-screw mechanism in lamellar microstructures. A Thompson’s tetrahedron indicated in the bottom as a reference…………………. 172

Figure 4.16. A schematic of the jog-screw dislocation motion looking down at the glide plane…………………………………………………………………………………………….. 172

Figure 4.17. The variation of the effective stress \( \tau_{\text{eff}} = \tau - \tau_b \) as a function of lamellar spacing…………………………………………………………………………………………………………………………………….. 173

Figure 4.18. TEM measurements of the dislocation density as a function of lamellar spacing…………………………………………………………………………………………………………………………………….. 173

Figure 4.19. A schematic showing the various parameters measured in a typical stress drop test…………………………………………………………………………………………………………………………………….. 174

Figure 4.20. The variation in instantaneous strain during a stress drop for the
different microstructures. Note the large strains for the FL microstructure. Also
indicated is the elastic strain during the stress drop calculated from a Young’s
modulus of 153 GPa…………………………………………………………………

Figure 4.21. The variation instantaneous strain during a stress jump for the
different microstructures. The strains for the FL microstructure is again the largest.

Figure 4.22. The ratio M’ for the different microstructures. The value of M’ is less
than 1 for all cases except one……………………………………………………… 175

Figure 4.23. The ratio R’ for the three PST orientations. The N orientation has an
R’ value almost an order of magnitude higher than the other two………………….. 176

Figure 4.24. The ratio R’ for the FL and equiaxed microstructures. FL
microstructure has an R’ value, an order of magnitude larger than that for the
equiaxed indicating large back stresses………………………………………………... 176

Figure 4.25. The ratio R’ for the FL is compared with that of the PSTs. FL
microstructure has an R’ value comparable to the N-orientation at strains
corresponding to minimum strain rate……………………………………………… 177

Figure 4.26. The anelastic strain for the three PST orientations compared. Trends
are similar to that observed for R’…………………………………………………… 177

Figure 4.27. The anelastic strain for the equiaxed and the FL microstructure is
compared. Trends are similar to that observed for R’………………………………. 178

Figure 4.28. The anelastic strain for the FL microstructure compared with that for
the three PST orientations. Trends are similar to that observed for R’……………... 178

Figure 4.29. Anisotropy in the creep behavior of the PST crystal is evident. The B
orientation creeps significantly faster than the two hard orientations………………. 179

Figure 4.30. Creep data from Parthasarathy et al comparing the primary creep
strains of the various microstructures. The FL polycrystal has primary strains
comparable to the 45° oriented PST [13]…………………………………………… 179

Figure 4.31. Data from Wegmann et al. suggests that at high stresses, the creep
rates and stress exponents of the FL polycrystal closely parallels that of the hard-
oriented PST crystal, while the low-stress exponent is close to that of the soft
oriented PST [15]…………………………………………………………………… 180

Figure 4.32a. The origin of backflow driven by the unzipping of the dislocation
segments along the interface……………………………………………………….. 180
Figure 4.32b. The backflow process is hindered when the channeling dislocation has jogs. This leads to a prolonged back flow behavior.

Figure 4.33. Creep data from Parthasarathy et al. suggesting that the FL polycrystal behaves like the 45° oriented PST [13].

Figure 4.34. The premise for modeling creep rates in fully lamellar structures with a distribution of lamellar spacing is that lamellae thinner than a critical cut off \( \lambda_c \) would not experience any effective stress and would not participate in the deformation process.

Figure 4.35. Schematic of the isostress averaging approach.

Figure 4.36. The log normal fit for the fine and coarse binary lamellar alloy. The fit parameters are indicated.

Figure 4.37. The model predictions are indicated along with the experimental values. Also shown are the experimental values and model predictions for the equiaxed microstructure. The model predicts the general trends of FL alloys, i.e., higher stress exponents, lower creep rates (compared to equiaxed) and the lamellar spacing effect.

Figure 4.38. The log normal fit and the fit parameters for the K5 alloy are indicated. The fit is much poorer for K5 alloys.

Figure 4.39. The model predictions and the experimental values for the K5 alloy are shown.

Figure 4.40. Data from Ott, suggest that there is an effect of alloying additions on the creep behavior of equiaxed microstructures. Solute effects have been ignored in our model [18].

Figure 4.41. Schematic of the interface sliding model suggested by Raj and Ashby adapted for lamellar microstructures (adapted from ref [23]).

Figure 4.42. The low and high stress predictions for the coarse lamellar binary alloy.

Figure 4.43. The low and high stress predictions for the fine lamellar binary alloy.

Figure 4.44. The low and high stress predictions for the K5 alloy.

Figure 4.45a. The cumulative strain rate for the entire stress regime for the coarse lamellar alloy (assuming interface sliding and jogged-screw mechanism to be independent processes).
Figure 4.45b. The cumulative strain rate for the entire stress regime for the fine lamellar alloy…………………………………………………………………………………... 188

Figure 4.45c. The cumulative strain rate for the entire stress regime for the K5 alloy…………………………………………………………………………………. 188

Figure 5.1. TEM micrograph of FL K5 alloy in the as-solutionised condition……... 214

Figure 5.2. TEM micrograph of FL K5 alloy after a 24 hr aging treatment at 900\degree C. Thinning and loss of $\alpha_2$ is evident…………………………………………... 214

Figure 5.3. Distribution of $\alpha_2$ lath spacings before and after aging……………….. 214

Figure 5.4. Distribution of $\gamma$ lath spacings before and after aging. Increase in the number of ultrafine $\gamma$ laths observed……………………………………………………... 215

Figure 5.5. a) Formation of ultrafine gamma laths that are arranged in packets, and b) Packet formation appears to take place by the growth of secondary gamma nuclei, as shown. The growth of one such gamma lath terminated by the B2 precipitate…………………………………………………………………………… 215

Figure 5.6. Precipitation of B2 phase associated with dissolving $\alpha_2$ laths………….. 216

Figure 5.7. TEM micrograph of FL K5SC alloy in the as-solutionised condition…. 216

Figure 5.8. Distribution of $\alpha_2$ & $\gamma$ lath spacings before and after aging. Increase in the number of ultrafine $\gamma$ laths observed in this case as well……………………….. 217

Figure 5.10. The creep behavior of the K5 and the K5SC alloy in the aged condition at the same stress level…………………………………………………… 218

Figure 5.11. The strain rate-strain plot for the above creep tests…………………… 218

Figure 5.12. Crept microstructures of the K5SC alloy indicate extensive interaction between the precipitates and the dislocations at the interface…………………….... 219

Figure 5.13. The different ways in which precipitation strengthening is manifested…………………………………………………………………………... 219

Figure 5.14. Strings of precipitates found in the middle of $\gamma$ laths. These precipitates help in obstructing the motion of dislocations within the $\gamma$ laths and this is seen in the frequent pinning and bowing of dislocations at these precipitates……………………………………………………………………... 220
Figure 5.15. The creep behavior of K5SC (aged) material is compared with K5SC (unaged) and K5 (aged) at 315 MPa and 760 °C. The K5SC aged alloy is superior to the other two……………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………………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Figure 5.30. Change in $\alpha_2$ volume fraction as a function of strain…………………. 227

Figure 5.31. $\alpha_2$ volume-fraction indicated as a function of time for the crept and the exposed samples………………………………………………………………… 227

Figure 5.32. Average $\alpha_2$ thickness indicated as a function of time for the crept and the exposed samples. The crept samples appear to retain “thicker” $\alpha_2$……………... 228

Figure 5.33. Formation of recrystallized gamma grains in the vicinity of ultrafine packet structure. Recrystallized grains as large as 10 microns have been observed... 228

Figure 5.34. The interfacial a) (111) plane of $\gamma$ and b) basal plane of $\alpha_2$ are indicated. The darker atoms are the Ti atoms, while the lighter atoms are Al. It is clear that both materials maintain stoichiometry along in these planes……….. 229

Figure 5.35. Lower minimum creep rate but poorer tertiary creep behavior observed for the N-orientation [22]………………………………………………………………….. 229

Figure 5.36. The difference in tertiary creep behavior between the two orientations is even more dramatic. [23]……………………………………………………………………. 229

Figure 5.37. Our results are identical to those obtained by the others. Lower minimum creep rate but poorer tertiary behavior for the N-orientation under compression………………………………………………………………………… 230

Figure 5.38. HREM image of Shockley type “transformation” ledge dislocations. Passage of one of these on every other interface plane (along with diffusion) causes $\alpha_2 \rightarrow \gamma$ transformation [27]…………………………………………………………. 230

Figure 5.39. HREM image of “misorientation” ledge probably similar to a Frank partial [27]…………………………………………………………………………... 231

Figure 5.40. Total volume fraction of $\alpha_2$ lost during creep may not be significant in the stabilized microstructures. However, $\alpha_2$ is often observed to be discontinuous (or spheroidized)…………………………………………………………………… 231

Figure 5.41. Strings of a) B2 precipitates in the K5 alloy and b) carbide/silicide precipitates in the K5SC alloy, retail the “lamellar” nature of the microstructure and prevent it from disintegrating completely……………………………………. 232

Figure 5.42. BF image (g=(111)) of K5 aged sample crept at 315MPa, 760C, 4.7% strain. Trans-lamellar twinning is seen at these large strains…………………. 232
Figure 5.42. BF image (g=(111)) of K5 aged sample crept at 315MPa, 760C, 4.7% strain. Coarsening of lamellar microstructure (effectively the lamellar spacing is widened) due to twinning and recession of $\alpha$. .................................................... 232

Figure 6.1. An incubation period is observed after a stress drop in the equiaxed $\gamma$-TiAl alloy. This behavior has been observed for different stress drops…………….. 241

Figure A.1. Furnace Heating Curve………………………………………………… 242

Figure A.2. Thermocouple calibration curve……………………………………….. 242

Figure A.3. LVDT Calibration Curve ............................................................ 242

Figure A.4. Load Cell Calibration Curve....................................................... 242

Figure B.1. Creep microstructure of Ti-6Al $\alpha$-Ti alloy indicating cusped screw dislocations…………………………………………………………………… 244

Figure B.2. The critical jog height variation is indicated. Predicted critical jog heights compare favorably with experimentally measured heights…………… 244

Figure B.3. The predictions of the jogged-screw model for $\alpha$-Ti is excellent, lending further support to the model………………………………………………... 244

Figure C.1. The isostrain-rate and isostress predictions are shown for the coarse lamellar binary alloy. The isostrain-rate assumption predicts a better fit than the isostress assumption………………………………………………………………… 246

Figure C.2. The isostrain-rate and isostress predictions are shown for the coarse lamellar binary alloy………………………………………………………………... 247
LIST OF TABLES

Table 1.1. Properties of TiAl alloys compared with Ti$_3$Al alloys and superalloys [2]................................................................................................................................. 45

Table 4.1. Schmid factor for each slip system (for ordinary slip) for a PST crystal oriented in the A (0°), B (45°) and N (90°) orientations (This calculation was made assuming that the Burger's vector is oriented at 45° to the stress axis). The Schmid factor is shown for all six $\gamma$ variants. The mode of slip (hard or soft) is also indicated. The darker shade of grey indicates the slip-system with the maximum Schmid factor. The lighter shade of grey is the slip system with the second highest Schmid factor. It is evident that for the soft orientation, true soft mode has the maximum Schmid factor in all variants but one. For the hard orientations, hard-mode deformation is strongly favored I most variants........................................ 189

Table 5.1. The matrix of exposure and creep tests conducted to probe the effect of temperature and stress on microstructural stability................................................. 233
CHAPTER 1. INTRODUCTION

1.1. Motivation

There has been a significant interest in γ-TiAl based titanium-aluminide alloys for the past couple of decades. These alloys possess a very low density (~4 g/cm³), high stiffness, good oxidation resistance, high thermal conductivity, moderate creep and high temperature strength [1, 2]. A comparison of the properties of gamma based alloys is compared with other high temperature alloys in Table 1.1 and Figure 1.1.[2]. These properties make these alloys attractive for high temperature structural applications, especially in the aerospace and automotive industries.

These alloys are just beginning to find their way into low risk static components. High specific stiffness of these materials can be exploited wherever clearances are a concern, for example, as valves, frames, seal supports, transition duct supports and cases. The higher stiffness also aids in shifting the vibration frequencies upward, which is beneficial for structural components. First generation gamma TiAl based alloys are being tested in the automobile industry in the form of turbocharger wheels and combustion valves [3]. The other perceived uses are in rotating and reciprocating high temperature components including high-pressure compressor stators and low-pressure turbine blades[4]. With careful alloy design, γ-TiAl appears to be capable of substituting for
superalloys (especially in the 600-800°C temperature range) with substantial weight saving and minimal redesign.

Creep resistance is of primary importance for structural applications involving extended exposure to high temperatures. Binary alloys have been found to have relatively poor creep resistance. Alloy development and microstructural design has lead to a significant improvement in creep behavior. However, as pointed out in a review article [5], the present understanding of creep mechanism(s) and the effect of alloying and microstructure is limited. This is despite the fact that there exists a large body of work done in the area of high-temperature creep. Clearly a difficulty is that creep is influenced by numerous microstructural parameters [6] and many of the investigations in γ-TiAl have been made on different structures. This lack of a fundamental knowledge of creep limits the potential of γ-TiAl as a high-temperature structural intermetallic compound. This also explains the lack of a satisfactory creep model in the literature. These gaps in the current knowledge of creep mechanisms (and the effect of microstructure and chemistry) have provided the motivation for this investigation.

1.2. Microstructural Characteristics

The binary Ti-Al phase diagram is shown in Figure 1.2. Gamma-TiAl based alloy systems typically contain 42-56 atomic percent aluminum. Stoichiometric and aluminum rich alloys (Al at % >50) are typically single-phase γ alloys. γ-TiAl is an ordered, intermetallic compound of Ti and Al with a L1₀ structure (face centered tetragonal) as shown in Figure 1.3 [2]. The lattice parameters are a=4.01 Å and c=4.07 Å. If we ignore the difference between a and c, we can think of gamma as an ordered fcc structure. Most
alloys of engineering interest are aluminum-lean and are two-phase alloys, the second phase being $\alpha_2$ (Ti$_3$Al) which is an ordered hexagonal compound (D0$_{19}$) as shown in Figure 1.4. The lattice parameters of $\alpha_2$ are $a=5.78$ Å and $c = 4.65$ Å.

$\alpha_2 + \gamma$ two-phase alloys have been seen to exhibit superior properties and have been the best studied and characterized of all gamma-based TiAl alloys. Controlled thermo-mechanical processing, heat treatments and cooling rates result in three major types of microstructures for the two-phase alloys: equiaxed, fully lamellar (FL) and duplex (Figure 1.5). The equiaxed structure consists of equiaxed $\gamma$ grains with $\alpha_2$ distributed as particles or as a semi-continuous layer at grain boundaries and triple junctions [7]. The fully lamellar structure consists of parallel laths of $\alpha_2$ and $\gamma$ phases forming colonies (usually > 150 $\mu$m), with little or no $\gamma$ grains in the colony boundaries [8]. Lamellar spacings typically range from 0.1 $\mu$m to 2 $\mu$m. A duplex microstructure consists of varying fractions of both equiaxed single-phase $\gamma$ grains and fully lamellar colonies.

The development of each of these microstructures is achieved via different heat treatments. The fully lamellar microstructure is achieved by solutionizing in the single-phase $\alpha$ phase field and subsequently cooling it. The transformation of prior alpha into variants of $\gamma$ and $\alpha_2$ in a plate form leads to a lamellar microstructure. Since the solutionizing temperatures are quite high, the prior-$\alpha$ grains are large and the resultant lamellar colonies have large grain sizes too. The typical grain (colony) size of a lamellar microstructure is 100-150 microns. The equiaxed microstructures can be achieved by solutionizing in the $\gamma$-phase field and rapidly cooling it back into the two-phase field.
However, solutionizing in the gamma phase field is available only for a small window of compositions (very close to stoichiometry). So most solutionizing treatments of aluminum-lean alloys are done in the $\alpha + \gamma$ phase field. Such a treatment results in the formation of a duplex microstructure (with the prior-$\alpha$ grains transforming into lamellar colonies). For a given composition, the volume fraction of alpha grains (in the $\alpha + \gamma$ phase field) is dependent on the solutionizing temperature. Temperatures closer to the gamma phase boundary lead to a smaller volume fraction of prior-$\alpha$ grains and the resultant duplex microstructure is closer to the equiaxed microstructure. Temperatures closer to alpha phase boundary lead to a large volume fraction of prior-$\alpha$ grains and the resultant duplex microstructure is dominated by lamellar grains rather than equiaxed. Since the solutionizing temperatures are lower, the grain size of the duplex microstructure is relatively small (of the order of 25-50 microns). It is evident from the phase diagram that microstructural control is possible not only by heat-treatments, but also by the control of the composition. The fully-lamellar microstructure has the best creep resistance (though duplex microstructure has been shown to have enhanced ductility)[9, 10]. It is thus important to understand the formation of the lamellar microstructure, since this will help design lamellar microstructures with enhanced creep properties.

The $\gamma$ and $\alpha_2$ lamellae in the FL microstructure are observed to have the "Blackburn" orientation relationship.

$$\{111\} \gamma \parallel (0001) \alpha_2 \text{ and } <110> \gamma \parallel <11\overline{2}0> \alpha_2.$$
The interface plane is parallel to the close packed planes in each phase, namely the (0002) basal plane in $\alpha_2$ and the $\{111\}$ in $\gamma$. There are six differently oriented $\gamma$ variants in the lamellar structure as the $<110>$ and $<101>$ directions in the L1$_0$ structure are not equivalent [11] (the notation for crystallographic directions follows the Hug convention, $<hkl>$ implies [$\pm h$, $\pm k$, $l$] or [$\pm k$, $\pm h$, $-l$]) Each of these variants is related by rotation of 60°. Adjoining gamma variants meet to form three different types of $\gamma$-$\gamma$ interfaces: Pseudo-twin (60° rotation), a 120°-rotational-twin (sometimes called variant interface) and a true or perfect twin (180° rotation), as shown in Figure 1.6 [12, 13]. These interfaces are mainly found along the close-packed habit planes. Even within a single $\gamma$ lath, the different variants can meet, forming order domain boundaries. These are thought to be formed by the independent nucleation, lateral growth and impingement of two different $\gamma$ variants. These order domain boundaries do not follow particular habit planes.

The transformation of $\alpha$ into $\alpha_2$ and $\gamma$ plates is the result of two factors a) the low misfit between the phases along the close packed planes b) the relative ease of transformation mechanism that leads to this orientation relationship. Even though the details of the transformation are not completely established, it is generally accepted that the $\gamma$ lamellae probably nucleates from stacking faults in the $\alpha$ matrix lying on the basal plane. The growth of $\gamma$ from $\alpha$ phase is facilitated by the subsequent passage of one of the Shockley partials bordering the stacking fault. Repeating this process at every other basal planes of the hexagonal matrix brings about the desired change in stacking necessary to achieve the change in crystal structure [14]. The change in chemistry is facilitated by
diffusional processes. It is often envisioned that the growth of the gamma plate occurs by the movement of ledges (of the Shockley type) on the $\alpha_2/\gamma$ interface. Since only one habit plane (the basal plane in the prior $\alpha$ grain) is available, a single orientation of the lamellar plates is obtained in one colony. The residual $\alpha$ trapped between the growing $\gamma$ lamellae then transforms into the ordered $\alpha_2$ phase below the eutectoid temperature. The rate of cooling through the $\alpha + \gamma$ phase field determines the amount of non-equilibrium $\alpha_2$ retained at room temperature and the lamellar spacing [1]. Faster the cooling rate, finer the lamellae [15].

Segregation, compositional inhomogeneities and inconsistencies in temperature gradients during actual processing and heat treatment, can often lead to microstructures other than the three described above. Zhang et al.[16] show that $\gamma$ laths can sometimes precipitate on some non-basal planes of the $\alpha$ matrix, even at very slow cooling rates. This gives rise to Widmanstätten structure. The formation of packet type lamellar microstructure dominated by APBs and true twin boundaries which are arranged in packets has been reported. This was observed when the cooling rate through the $\alpha + \gamma$ phase field is large. This large variability in microstructure makes the study of these alloys significantly more complicated. A sound understanding of these features is crucial in resolving the variability of mechanical properties and for continued alloy development. The effect of alloying additions on the mechanical and physical properties is briefly explored in the next section.
1.3. Effect of chemistry.

Varying the aluminum content can lead to single phase $\gamma$-alloys (Al content at or above stoichiometry), or two-phase $\alpha_2 + \gamma$alloys (below stoichiometry). The $\alpha_2$ volume fraction increases with decreasing Al content. In the range of compositions from 43 to 53 at. % Al, the lowest strength occurs at compositions around 51 at. % Al. Room temperature ductility also varies with the aluminum content, exhibiting a maximum ductility around the two-phase composition of Ti-48Al [17]. In single-phase $\gamma$ alloys, the creep rates were found to increase as the Al content was increased [18]. The effect of aluminum content in the Al-lean side of stoichiometry is mostly related to the volume fraction of the second phase.

While most early studies were based on the binary alloy with 48 at% Al significant research has gone into developing ternary and quarternary alloys, particularly with the aim of bettering the high temperature properties [19]. The addition of an alloying element can also be beneficial for the control of microstructure and for improvement of ductility [20]. Yet another motivation for alloying has been to improve the high temperature oxidation resistance (additions of Cr, Mn and Si, in particular)

There is overwhelming evidence that in small quantities V, Cr and Mn significantly enhance the ductility of two-phase gamma alloys [17, 21, 22] (though, some claim these additions have no effect on ductility [2, 23]). The reason behind the favorable effect on ductility has been reasoned to be either because of phase stabilization or due to the nature of site occupancy. Cr, Mn, V have been seen to partition preferentially to $\alpha_2$ [19]. It has been suggested that stabilization of $\alpha_2$ (by these alloying
elements) is particularly beneficial since $\alpha_2$ acts as an ideal scavenger of interstitial impurities such as boron, oxygen and nitrogen, which would otherwise cause embrittlement of the gamma phase [17, 24]. It has been suggested that Mn and Cr decrease the covalent nature of bonding in $\gamma$-TiAl [25-27]. This is reported to reduce the tetragonality of the L1$_0$ structure and cause an increase in the number of deformation modes [27].

The addition of several alloying elements including Nb, Zr, Mo, V, W, Hf, Sn and Ta has been seen to increase the creep resistance [17, 28]. This is thought to be due to the solid solution strengthening of the gamma phase [17]. It has been proposed that the addition of W and Ta (and to a limited extent Nb) significantly slows down diffusion in the gamma phase [13]. These slow diffusing species also tend to stabilize the fully lamellar microstructures by slowing down lamellar coarsening processes (loss of metastable $\alpha_2$ laths during creep requires the repartitioning of these elements and this is slowed down). It has been proposed that W additionally lowers the stacking fault energy and this makes dislocation recovery mechanisms more difficult [1, 8, 19].

Si is seen to enhance the high temperature oxidation resistance [29]. In small quantities, it is also an excellent solid solution strengthener. Higher levels of Si has been seen to cause precipitation of silicides which leads to significant strengthening [30]. Si has also been credited for the retardation of dynamic recrystallization and microstructure refinement [29-31]. In the lamellar microstructures addition of Si has been reported to change the misfit between $\alpha_2$ and $\gamma$ and leads to the formation of interfacial misfit screw dislocation networks [32]. As a downside, the addition of silicon has been reported to be severely detrimental to the fracture toughness.
Cr, V, Nb and Mo have been seen to stabilize a third phase, the B2 phase. B2 is an ordered body centered cubic phase with CsCl structure. The B2 phase has a Ti/Al ratio close to that of $\alpha_2$, but contains 10-15% bcc stabilizing elements. In alloys containing these alloying additions, the lamellar microstructure often consists of composite $\alpha_2$+ B2 laths lying between the $\gamma$ lamellae. B2 is also seen precipitated at the colony boundaries in FL alloys and at grain boundaries in equiaxed alloys, after annealing [33]. The presence of the B2 phase has often been suggested to improve room temperature ductility, but adversely affect creep properties. This is in part due to role played by the B2 phase in causing grain refinement.[34]. On the contrary, the role of the B2 phase lying along the lamellae has been suggested to cause precipitation-hardening [8]. It is obvious that the role of this phase on the creep behavior is not clear.

Interstitial elements such as C, B, O and N have been shown to affect the mechanical properties significantly [19, 35, 36]. Carbon and Nitrogen containing alloys have been shown to cause extensive precipitation hardening and show typical age-hardening behavior [35, 36]. Solid solution strengthening via solute drag has also been proposed as a possible strengthening mechanism for carbon, particularly in FL microstructures [10, 35]. Both Carbon and Boron additions have been credited with grain size refinement [27, 35]. In the case of boron, precipitation of plate like borides from the melt and during aging has been suggested as a reason for microstructural refinement [37]. Boron addition is also seen to promote the formation of duplex and equiaxed microstructures, and cause discontinuous $\alpha_2$ and packet structure of fine $\alpha_2$ lamellae in FL microstructures. These microstructural effects are generally regarded detrimental to the creep properties.
Manipulation of the microstructure and alloy design has lead to the general improvement of both room temperature and high temperature properties. However, a clear understanding of the mechanisms involved is still warranted, such that the scope for predictability of the various mechanical and physical properties can be expanded. In the following section, general creep mechanisms in metals and alloys will be described. In subsequent sections the current knowledge of deformation in γ-TiAl based alloys will be reviewed with an emphasis on the creep behavior.

1.4. Creep deformation in metals:

Time dependent plastic deformation typically at high temperatures is referred to as creep. A creep test involves applying a constant load or stress to a sample, while monitoring the change in its dimensions. The accumulation of strain with time is graphically represented by the creep curve [Figure 1.7]. This strain is usually divided into three stages. The first stage is called primary creep (usually after an instantaneous plastic strain) and this typically involves an ever-decreasing creep rate that is associated with strain hardening. The second stage is called steady-state or secondary creep during which the creep rate remains more or less constant. This is so because the strain hardening mechanisms are more or less offset by recovery mechanisms. The third and final stage is called tertiary creep, which involves an accelerating creep rate, which may terminate in fracture in the case of tensile specimens. At this stage recovery and damage processes dominate leading to the weakening of the microstructure.

There are many variations to the shape of the creep curve. At low temperatures, the recovery mechanisms are relatively sluggish and so only primary creep transients are
observed. These ever-exhausting curves can usually be described using a logarithmic law, or a power law with time exponents close to 1/3. In some cases (and at higher temperatures), tertiary creep commences as soon as primary creep ends and the steady state creep rate is usually replaced by a minimum creep rate. Depending on the deformation mechanism it is also possible to observe an inverse primary creep, where the creep rate actually increases in the initial stages.

There are several measures of a material's creep strength. The commonly used parameters are the primary creep strain, the time to failure, the rupture strain and most commonly, the steady-state creep rate. The steady state creep rate gives a good measure of the underlying hardening and recovery mechanisms. This stage of creep usually lasts for many orders of magnitude in time and it is assumed that the sub-structure remains essentially unchanged during this period. The steady-state microstructure has been found to be very sensitive to stress and quite insensitive to temperature.

1.4.1. Strain rate dependence on temperature and stress

High temperature creep is a thermally-activated process. There is substantial evidence to indicate that the steady state creep rates have an Arrhenius type dependence on temperature.

\[
\dot{\varepsilon}_s = \dot{\varepsilon}_0 \cdot \exp\left(-\frac{Q_c}{RT}\right)
\]  

(1.1)

where \(\dot{\varepsilon}_s\) is the steady state creep rate, \(\dot{\varepsilon}_0\) is a pre-exponential term, \(Q_c\) is the activation energy for creep, \(R\) is the gas constant and \(T\) is the temperature. It is also well established
that the dependence of the self-diffusion coefficient \( D_L \) on temperature is through an Arrhenius type equation of the type:

\[
D_L = D_0 \cdot \exp\left(-\frac{Q_d}{RT}\right)
\]  

(1.2)

where \( D_0 \) is a pre-exponential term, \( Q_d \) is the activation energy for self diffusion,

It has been found that for most materials [38], at high temperatures, \( Q_c \approx Q_d \). This is not a surprising result since atomic mobility is very large and can facilitate numerous dislocation recovery processes. In fact, even purely diffusional processes could cause creep at these temperatures. At lower temperatures, short-circuit diffusion paths along dislocation cores and grain boundaries become more favorable than bulk lattice diffusion. In this low temperature range (0.25 to 0.5 \( T_m \)), the creep activation energy \( Q_c \approx Q_{g.b} \approx Q_{pipe} \approx Q_d/2 \). It is thus logical to assume that the steady state creep rate is proportional to the \( D_{eff} \), where \( D_{eff} \) is the effective diffusion coefficient due to lattice, pipe and grain boundary diffusion [39].

For most materials, it has also been shown that the steady-state or minimum creep rate can be related to the applied stress via a power law. These empirical observations can be combined in a single equation, generally known as the Dorn equation [39]:

\[
\dot{\varepsilon}_s = A \cdot D_{eff} \cdot \left(\frac{\sigma}{G}\right)^n
\]

(1.3)

where \( A \) is a constant, \( D_{eff} \) is the effective self-diffusion coefficient, \( \sigma \) is the applied stress, \( G \) is the shear modulus and \( n \) is termed the stress exponent. For a given material, the \( n \) value is found to be constant over a range of stresses. Though this equation is purely empirical, the values of the stress exponent \( n \) and the creep activation energy \( Q_c \) are
thought to provide a phenomenological understanding of the underlying creep mechanism. In the following section we will briefly review the common high temperature creep mechanisms.

1.4.2. Creep Mechanisms

a) At low stresses and temperatures close to the melting point, materials have been observed to flow in a Newtonian viscous manner. Creep at these stress levels does not involve dislocations but matter transport that is purely diffusion-mediated. The mass flux is caused by gradients in vacancy concentration that are set up by the different stress states at different grain facets. The type of diffusional creep involving lattice diffusion is commonly called Nabarro-Herring (NH) creep [40], [41]. The stress exponent for this mechanism is unity and the activation energy corresponds to that of lattice diffusion. At lower temperatures, diffusional creep process involving grain boundary diffusion becomes predominant. This mechanism, called Coble creep, again predicts a stress exponent of unity and an activation energy that corresponds to that of grain boundary diffusion [42]. Accounting for grain boundary sources and sinks of vacancies can give rise to a stress exponent of 3 [39]. These mechanisms require a concomitant sliding of grain boundaries so that compatibility between grains is maintained. This sliding is called intrinsic or Lifshitz sliding. Atomic flux is dependent on the grain size. Hence the strain rate produced due to these mechanisms is inversely proportional to square or cube of the grain size for NH and Coble creep respectively. Larger grain sizes are thus beneficial for minimizing the contribution of diffusional creep mechanisms.
b) When stresses are higher, the contribution of dislocations to creep strain becomes significant. The behavior of solute-strengthened alloys and pure metals is observed to be quite different. Solute strengthened alloys in general exhibit a stress exponent of about 3. The creep curve indicates no primary creep behavior (in fact, inverse primary creep is observed frequently). It has also been observed that the activation energy corresponds to the lattice diffusivity of the solute species. The development of dislocation substructure is hardly evidenced. These observations point towards a mechanism that involves the viscous glide of edge dislocations, where the dislocation mobility is limited because of solute drag. Alloys that behave in this manner have been termed as "Class I" alloys [38]. The stress exponent of 3, also called the "natural third power law" is not restricted to a solute drag mechanism. A mechanism involving the glide of screw dislocation that carries several unit jogs along its length, gives rise to a natural third power law [39]. Even a dislocation climb mechanism such as Nabarro creep can give rise to the natural third power law [43]. These latter two mechanisms have an activation energy that corresponds to that for lattice self-diffusion. The universality of the third power law comes from the following equations which is obeyed by the above mechanisms:

\[ \dot{\varepsilon} = v \cdot \rho \cdot b \]  
\[ \rho = \left( \frac{\tau}{\alpha \cdot G \cdot b} \right)^2 \]  
\[ v \propto D \cdot \tau \]  

where \( \dot{\varepsilon} \) is the strain rate, \( \rho \) is the dislocation density, \( b \) is the Burger's vector, \( v \) is the dislocation velocity and \( \alpha \) is called the Taylor's parameter. All other parameters have
been previously defined. Combination of equations (1.4), (1.5) and (1.6), gives rise to the natural third power law.

c) Pure metals and some solute-strengthened alloys (Class M alloys) show a significantly different behavior than the Class I alloys. The stress exponents observed are around 5. In stark contrast to Class I alloys, these alloys show significant primary creep. Microstructural observation indicates the formation of subgrains. The size scale of the substructure has been seen to be inversely proportional to the applied stress. Depending on the temperature, the activation energy has been seen to correspond to either lattice diffusion or pipe diffusion. The fundamental understanding of creep mechanisms in these alloys has been less satisfactory. Weertman proposed that dislocation production at sources is limited by the climb of dislocations at the tip of pile-ups [44, 45]. Unfortunately, pile-up structures are rarely seen in high temperature creep microstructures. What is observed, though, is the formation of subgrains, which are relatively free of dislocations and subgrain boundaries, which consist of dense dislocation networks. A model based on these "soft" and "hard" regions has been proposed [46]. In a more recent approach creep in pure metals has been modeled as an interaction of thermally activated obstacle bypass, substructure refinement due to increase in dislocation density and the substructure coarsening due to diffusion aided recovery processes [47]. Though the model does not delve into the detailed mechanics of each of these coupled processes, its applicability appears to be universal to recovery controlled creep processes. The need for a marriage between physically-measurable microstructural parameters and phenomenological models is evident.
d) A few other creep mechanisms will be briefly reviewed here. A dislocation creep mechanism called "Harper-Dorn" creep is often reported (especially at high temperatures and very low stresses) [48]. The stress exponent predicted is unity. Though the mechanistics of H-D creep is still debated, it appears that this mechanism has no apparent grain size dependence and thus is different and unique from Nabarro-Herring creep. It also appears that the Taylor's relation whose validity is generally accepted, does not seem to be applicable in H-D creep. "Grain boundary sliding" is often considered to be an independent creep mechanism [39]. As mentioned earlier diffusional creep (N-H or Coble creep) requires that grain boundaries slide. It has been shown that it is equivalent to imagine the same scenario as grain boundary sliding being accommodated by diffusion (Intrinsic sliding). However, accommodation can also be achieved by plastic deformation and these sliding mechanisms have been referred to as Rachinger or extrinsic sliding. The stress exponent predicted is close to 2. In dispersion strengthened materials, a high stress exponent (8 or higher) is often reported and this has been related to the existence of "threshold stresses", that has to be overcome to cause slip [39]. This threshold has been shown to be related to the stress required to overcome dislocation-particle interaction (this may be due to local or general climb of edge dislocations, cross -slip of screw dislocations or the break away of dislocations from attractive incoherent particles)
1.5. Deformation in Gamma TiAl

Many properties of γ-TiAl have been attributed to the nature of bonding. The Ti-Al bonds are strong and covalent. The consequences of this is the fact that γ remains ordered to melting point (~1440°C). It results in high stiffness that is well retained even at very high temperatures. The strong bonding also leads to a high barrier to diffusion. In addition, this leads to a large anti-phase domain boundary (APB) energy. The high elastic modulus and high diffusion barrier make γ-TiAl alloys quite creep resistant. However, for the same reasons, γ-TiAl is quite brittle (a consistent room temperature tensile ductility over 2% has not been reported, even for the most ductile, duplex microstructure alloys). The low ductility results in low fracture toughness and poor fatigue properties. Stress concentrations, unpredictable loading conditions and impact loading (say, due to foreign or domestic object damage in aircraft engines) can even lead to catastrophic failure. Ductility is essential from a processing point of view as well. Processing parameters required for crack-free casting and forging routes, are extremely stringent and manufacture of large parts becomes expensive. Hence it is crucial that the general characteristics of plastic deformation be understood, before delving into the details of creep deformation.

1.5.1. Common deformation modes in γ-TiAl

As in closed packed cubic structures, slip occurs on the {111} planes in closed packed directions. The 1/2[110] dislocations are ordinary perfect dislocations (the lowest energy perfect dislocations) and have been referred to as "easy slip" dislocations [Figure 1.8]. The [101] and [011] dislocations, which are equivalent, are the higher energy
superdislocations. The $1/2[112]$ dislocations are also superdislocations on the close packed planes. Slip systems on planes that are not close packed, such as $<100>[110]$, are active only at very high temperatures and will not be discussed.

Various dislocation dissociation reactions can take place on ordinary and super dislocations and these will create partial dislocations. Ordinary dislocations can dissociate into two Shockley partials. However, due to the nature of ordering in $L1_0$ compounds, the Shockley partials are separated by a complex stacking fault (CSF) instead of a simple intrinsic stacking fault.

$$1/2 [\bar{1}10] \rightarrow 1/6 [\bar{2}11] + CSF + 1/6 [\bar{1}2\bar{1}]$$

The $[011]$ superdislocation can dissociate into two $1/2[011]$ partials that are separated by an antiphase boundary (APB).

$$[011] \rightarrow 1/2 [011] + APB + 1/2 [011]$$

The $1/2[011]$ can further dissociate into Shockley partials. Again, owing to the nature of $L1_0$ ordering the fourfold dissociation gives rise to bordering stacking faults of different nature [49]. The total reaction is shown below.

$$[011] \rightarrow 1/6 [\bar{1}21] + CSF + 1/6 [112] + APB + 1/6 [\bar{1}21] + SISF + 1/6 [112]$$

SISF is a superlattice intrinsic stacking fault. The SISF energy is much smaller than that of the CSF. So the dissociation is often threefold [49]. The $1/2[112]$ superdislocation can undergo two dissociation reactions:

$$1/2 [112] \rightarrow 1/6 [112] + SISF + 1/6[112] + SESF + 1/6 [112]$$

$$1/2 [112] \rightarrow 1/2 [010] + APB + 1/2[011]$$

These $1/2[101]$ and $1/2[011]$ further dissociate in reactions similar to those described earlier.
Besides slip, twinning is the other major deformation mode. Twinning in γ-TiAl occurs through a $1/6[\bar{1}\bar{1}2]$-type shear vector on the ($\bar{1}11$) twin plane. This shear vector creates what has been termed a "true-twin". Wang et al. [50] have shown that this type of twinning can be achieved by successive cross-slip of the twinning dislocations $1/6[\bar{1}\bar{1}2]$ on the ($\bar{1}11$) plane that leave $1/6[011]$ stair-rod dislocations on the (100) plane. Twins are typically seen to nucleate from grain boundaries [51, 52]. Twins can also nucleate within the grain interior by a pole mechanism involving a Frank partial dislocation turning around a perfect dislocation [7, 53, 54].

von-Mises compatibility criterion requires five independent slip (or twin) systems and this seems to be the reason why deformed microstructures show several types of dislocations and twins (due to the inherent anisotropy of ordered γ-TiAl). The exact choice of the deformation modes appears to depend on the nature of the dislocation core and fault energies, and these are very sensitive to temperature and composition. In general, at low temperatures ordinary dislocations, $<101>$ superdislocations, and $1/2[112]$ superdislocations (that appear to be dissociated locally to form faulted dipole loops bounded by Shockley partials) are seen [49]. At higher temperatures the deformation microstructure is dominated by $1/2[110]$-type ordinary dislocations [55-59]. The dislocation type is also strongly affected by alloy chemistry. Al-lean binary alloys have been seen to have twins and $1/2[110]$ ordinary dislocations, while the Ti-lean [49], 52-Al alloys have superdislocations and dipoles (these were deformed at room temperature).

Like L1$_2$ alloys, γ-TiAl also exhibits a strong flow stress anomaly. Yield stress is seen to be more or less constant upto 600°C above which it increases with increasing temperatures and peaks at around 800°C [Figure 1.9][56, 60, 61]. Veysierre et al. have
suggested that the anomaly is the result of the formation of Kear-Wilsdorf (KW) locks [49]. These locks are formed by the dissociation of the <011> superdislocation on the {111} planes. The resultant Shockley partials can cross slip on the {111} planes. However, the energy of the APB formed is minimized along the {001} plane. This leads to a non-planar dissociation configuration that is essentially sessile. This is called the KW lock (similar non-planar configurations have also been credited for the tension-compression asymmetry [62]). However, the observation of primarily ordinary dislocations at high temperatures doesn’t substantiate the formation of KW locks. It has been suggested that the increasing number of pinning points (jogs) on ordinary screw dislocations (due to frequent cross-slip processes) is the source of yield strength anomaly [55, 56]. High CSF energies have been calculated in γ-TiAl [12] which leads to the compact nature of the screw dislocation, that is prone to frequent cross-slip. This has also been confirmed in HRTEM studies [63]. These observations seem to hint that the "jogged-screw" dislocation hypothesis may be valid.

1.5.2. Deformation in lamellar two-phase alloys

Two-phase alloys have been shown to exhibit a higher ductility than single-phase alloys. Many explanations have been provided for this observation, yet a clear picture has not emerged. Deformation in TiAl seems to be highly sensitive to the oxygen content. It has been suggested that reducing the oxygen content enhances ductility via the enhanced activity of 1/2[110] dislocations. The brittleness of γ-TiAl with oxygen has related to the enhancement of bond directionality due to oxygen [24]. Yet others offer that the lack of
ductility is due to precipitation of brittle oxide phases [64]. It seems most likely that that \( \alpha_2 \) acts as a scavenger/getter of interstitials [24].

The deformation response of individual colonies depends on the lamellar orientation with respect to the stress axis. Colonies with lamellae that are parallel or perpendicular to the stress axis are referred to as being in the "hard orientation" and the deformation mode is referred to as "hard-mode". The hard mode deformation involves shear that is translamellar (not parallel to the lamellar interfaces) and takes place by slip or twinning. The lamellar interfaces act as barriers to twin and slip transmission and consequently, this orientation is stronger and less ductile [8]. The yield strength has been reported to be related to the lamellar spacing through a Hall-Petch type relation.

Grains with lamellae oriented between 30-70° to the stress axis are in the "soft" or "easy" orientation. The soft-oriented grains are dominated by the so-called "soft-mode" deformation that takes place by shear parallel to the lamellar interface involving twinning and slip. The mean free path for dislocation movement is related to the domain size or colony size, which is two to three orders of magnitude larger than the lamellar spacing and this makes this orientation ductile and weak.

Colony boundaries can be completely eliminated in single colony samples and these are referred to as Poly-Synthetically Twinned (PST) crystals. These crystals represent the fundamental unit of the lamellar microstructure and are usually prepared using an induction float-zone method [65]. The orientation of the PST crystal is characterized by the angle \( \phi \). This is the angle subtended by the lamellar plates relative to the stress axis. \( \phi = 0^\circ \), referred to as the "A orientation", and \( \phi = 90^\circ \), referred to as the "N
orientation", are the hard orientations. Samples with $\phi \sim 35-55^\circ$ are referred to as B oriented and these are soft oriented [Figure 1.10]. Mechanical properties of PST crystals are extremely anisotropic [Figure 1.11]. Yamaguchi and coworkers [28] have observed that the N oriented PST crystals are up to five times stronger than the B oriented PST crystals. The yield stress of the A oriented PST lies midway between the N and B oriented PSTs. This plastic anisotropy is unique to lamellar structures; equiaxed single-crystal gamma does not exhibit this [65]. The analytical model of Hazzledine and co-workers attributes this flow anisotropy to interfacial strengthening [65]. Using a pile-up model, they have not only been able to model the flow stress dependence on orientation, but also the work hardening characteristics. Parthasarathy et al have extended this model accounting for the distribution of lamellar spacings [65]. They have also found that the yield behavior of FL microstructures seems to be dominated by the soft oriented grains at small strains and by the hard oriented grains (the A orientation) at larger strains.

There appears to be some confusion regarding the use of terms, hard and soft mode. While several authors consider soft-mode deformation to essentially involve shear parallel to the lamellae, yet others consider the deformation mode to be soft, only if the slip system is parallel to the lamellar interface. Screw dislocation lying on the interface plane (Burger's vector lies parallel to the interface plane) can cross slip onto the $\{111\}$ plane oriented across the lamella. These dislocations have also been regarded as hard mode dislocations by Wegmann et al, since these cause work hardening via a Hall-Petch effect [66]. However, it should be remembered that deformation of hard-oriented PST (say, an N or A oriented) crystal, does not geometrically require dislocations with Burger's vector in the interface plane. For this reason, throughout this thesis hard-mode
dislocations are those which have a Burger's vector that does not lie parallel to the lamellar interface. Soft mode dislocations gliding on non-interface planes will be termed trans-lamellar soft-mode dislocations.

The deformation of $\alpha_2$ is also an important issue. Weizorek et al have shown that $\alpha_2$ is non-participative in deformation at room temperatures. However, at high temperatures, $\alpha_2$ phase seems to deform involving dislocations with a and c components [67]. Slip and twin transmission from one $\gamma$-lath to the adjacent $\gamma$-lath via the sandwiched $\alpha_2$, has been observed in constant strain rate tests [67]. The $\alpha_2$ volume fraction in stabilized FL Ti-48Al is 5-8% and so the contribution of $\alpha_2$ deformation to the total strain is probably very small.

In the next section we will review the work done on the high-temperature creep behavior of single- and two-phase TiAl alloys

1.5.3. Creep deformation

It is acknowledged that the high temperature deformation mechanism can be quite different from the low-temperature constant strain-rate behavior. The creep behavior of gamma-based TiAl alloys has been of interest to a large number of researchers in the past decade. Many attempts have been to describe the phenomenology of creep and in ascribing mechanisms to the underlying creep processes based on stress exponents and activation energy. Only recently has attention been diverted to understanding creep behavior in terms of mechanistics. This section will summarize broadly both the phenomenology and the current understanding of creep mechanisms.
In general, creep deformation in single and two-phase alloys involves a normal primary transient followed by a very short secondary creep region, followed by tertiary creep associated with damage accumulation [68]. Though the shape of the creep curve for $\gamma$-based alloys is "typical" for all microstructures, there are differences that should be pointed out.

a) The FL microstructure has been consistently reported to have a superior creep resistance than both the duplex alloys and single-phase gamma alloys. The minimum creep rate of a two phase FL structure is usually more than an order of magnitude lower than the minimum creep rates of single phase TiAl and Ti$_3$Al as well as duplex structures tested at the same temperature and stress [25, 69, 70]. (Figure 1.12). The creep resistance differential between FL and duplex microstructures is thought to originate from the impediment caused by the lamellar interface to dislocation movement in the $\gamma$-laths.

f) FL microstructures seem to exhibit significant primary creep strains that are much larger than that of duplex and equiaxed microstructures [31]. Even the "instantaneous" plastic strains accumulated during loading can be significant. This has been reasoned to be due to the abundance of $\alpha_2/\gamma$ interfaces that act as dislocation sources [8, 71, 72].

g) Creep curves for the FL structure has been seen to harden very rapidly at the end of the primary creep stage [71, 73].

h) The FL microstructure has also been reported to exhibit tertiary creep under compression.
For γ-TiAl alloys, minimum creep rates are reported more often than steady state creep rates. Figures 1.13 and 1.14 represent a compilation of minimum strain-rate versus stress data for different microstructures. Dorn equation (Equation 1.3) is usually applicable at least over small ranges of stresses. While it is generally found that equiaxed and duplex microstructures exhibit a stress exponent close to 5, the FL microstructures seem to exhibit two different regimes in stress: a low stress regime where the stress exponent is between 2 and 3 and a high stress regime where the stress exponent is 8 or higher. In fact, Morris, Hayes and others have observed extremely high stress exponents of the order of 19-23 at high stresses [68, 71], but these stress exponents appear to be related to power-law breakdown.

The value of the activation energy Q has been found to be in the range of 280-320 kJ/mol by numerous researchers [8, 14, 23]. The activation energy for lattice self-diffusion in TiAl has been reported to be in the ballpark of 290-305 kJ mol⁻¹ [74]. It is clear that the creep mechanism (at least at high stresses) appears to be diffusion-mediated. There is, however, a large scatter in the activation energy data, with reported values as high as 600 kJ/mol and as low as 140 kJ/mol. This scatter probably originates from the fact that most of these results come from temperature change experiments. Lu and Hemker have found that temperature change experiments and a series of single temperature tests lead to vastly different values of activation energy [75]. The other source of this scatter is the effect alloying elements. The addition of elements such as Ta, W and Nb has been seen to increase the creep activation energy.

Purely on the basis of stress exponents and the creep activation energy, it has been suggested that creep in duplex and equiaxed microstructures is controlled by climb or
recovery processes [8]. The low stress exponents (and the smaller activation energy) at low stress in the FL microstructures has been attributed to diffusional creep mechanisms [68]. It has been proposed that the larger stress exponents at large stresses in FL microstructures arise due to what is called "constant structure creep" [8]. It is usually described by the following equation:

\[ \dot{\epsilon}_s = B \cdot D_{\text{eff}} \cdot \lambda^3 \cdot \left( \frac{\sigma}{G} \right)^8 \]  

(1.7)

where \( B \) is a constant, and \( \lambda \) is the subgrain size. This is essentially a dislocation climb controlled creep mechanism involving the formation of subgrains. Typically the subgrain size is inversely proportional to the applied stress and so the stress exponent is 5. In a lamellar structure, it is possible that the subgrain size is larger than the lamellar width, so \( \lambda \) takes the value of the lamellar spacing and is constant. There are two strong arguments against this creep mechanism. The first is that, this mechanism actually implies that when stresses become large enough, the subgrain size may become smaller than the lamellar size and this would cause the stress exponent to drop back to 5. However power-law breakdown is typically seen at large stresses. Secondly, the equation leads to a very strong lamellar spacing dependence \((\lambda^3)\). Such a strong lamellar spacing dependence has rarely been observed.

Alternately, the high stress exponent values at high stresses have been thought to originate from inhomogeneous deformation between the various lamellar colonies and the various lamellae within a colony [8, 76]. It has also been suggested that large stress exponents represents a mechanism of deformation in which internal stresses/back stresses
build up and a threshold needs to be overcome before the dislocations can become mobile [8, 71, 76].

It is clear that these hypotheses will remain purely phenomenological unless microstructural evidence is provided in their support. A limited number of studies have involved detailed analysis of deformation substructure developed during creep. The current knowledge of creep mechanism will be briefly summarised in the next section.

1.5.3.1. Creep mechanisms

As mentioned in a previous section (Section 5.2.), there are two principal modes for plastic deformation: slip and twinning. At high temperatures, shape change can be effected solely by diffusion too (NH creep). The real question is to determine which process becomes rate controlling (not necessarily the one which accounts for most strain).

Deformed microstructures of single-phase gamma alloys reveals ordinary and superdislocation activity and the formation of subgrains [8]. Two-phase alloys in duplex and equiaxed microstructures have been frequently shown to contain elongated 1/2[110] screw dislocations with pinning points and cusps along the length [31, 75, 77, 78]. Superdislocations and faulted dipoles have been occasionally observed too [79]. Dislocation tangles and subgrain formation are seen only at large strains [10, 79].

FL lamellar alloys deformed at high stresses have microstructures very similar to that of equiaxed and duplex alloys. The microstructure is dominated by cusped ordinary dislocations in the screw orientation. These dislocations are of both the hard and soft mode types. However, the reduction of the minimum creep rate for finer lamellar spacings, has been taken as evidence for the predominant role of hard-mode dislocations
in the $\gamma$ matrix. Slip transfer across the $\alpha_2/\gamma$ interface is difficult and rarely observed. Bartholomeusz et al. have also observed and indicated what appear to be an emission of dislocations from the $\gamma - \alpha_2$ interface into the adjacent $\gamma$ phase [69]. Other researchers have observed bowed out segments of ordinary dislocations emanating from the $\alpha_2/\gamma$ interfaces (these contain dislocation networks which act as sources for new dislocations) [71]. The glide of interfacial Shockley partials on the $\alpha_2 - \gamma$ interface has been reported as a significant deformation mode [26, 50, 80]. Such glide of interfacial dislocations could provide a mechanism for interface sliding at lamellar boundaries [81]. Fiducial line studies however suggest that the contribution of interface sliding to strain may be marginal [82].

Mechanical twinning is common in $\gamma$-TiAl even at high temperatures and at the low strain rates corresponding to creep conditions. The extent of twinning is seen to be significantly larger for equiaxed microstructures than FL microstructures [83]. This is thought to be related to von-Mises criteria due to which twinning becomes an essential deformation mode in the equiaxed microstructures, but not in lamellar microstructures [84]. The amount of twinning has been seen to increase with strain and stress [7, 51, 73]. However it has also been shown that mechanical twinning can occur even within 0.2-0.5% strain [7]. The total contribution of twinning to macroscopic strain is usually smaller than that due to slip and its role as a rate-controlling mechanism is quite unclear.

There are opposing views to the effect of twinning on microstructure evolution during creep. In equiaxed microstructures, twinning has been suggested to cause strain hardening (by microstructure refinement) [26, 37, 80]. On the contrary, twinning also
reorients part of the crystal favorably for slip, which would result in softening [7]. It is evident that the role of twinning is creep is still an open issue.

The tertiary creep behavior has been observed to be related to microstructural degradation due to the loss of $\alpha_2$ and its spheroidization (in lamellar alloys), inhomogeneous deformation, formation of shear bands, dynamic recrystallization, interlamellar and intergranular cracking and void formation [8, 31, 76, 85]. The possible effect of the environment has not been well addressed. Exposure to air at high temperatures could lead to embrittlement and the formation of surface cracks.

In the following section the microstructural factors affecting creep will be briefly reviewed. The effect of alloying additions has already been reviewed in Section 1.3.

1.5.3.2. Microstructural factors affecting creep in FL microstructures

a) Effect of lamellar spacing

It has been consistently reported in the literature that a finer lamellar structure provides lower creep rates [23, 27, 31, 86]. There are two interpretations for the lamellar spacing effect. First, the lamellar spacing determines the mean free path for mobile dislocations and provides strengthening in a typical Hall-Petch fashion. Second, the interfaces cause the dislocation segments to bow between them and the bowing stress is inversely proportional to the lamellar spacing.

Some researchers have reported that the creep rate is relatively independent of lamellar spacing [54, 86]. Yet others suggest that there is an optimum lamellar spacing and very fine lamellar structures cause weakening [53]. Studies by Yamamoto et al.
indicates that fine lamellar spacings (0.1µm) are beneficial at high stresses, but could cause weakening at low stresses [86]

**b) Colony size and boundary effects**

Huang et al.[2] propose that the superior creep resistance of the lamellar structure is primarily an effect of its large grain size but the conclusions are not based on a systematic study taking into account the effect of other factors, while keeping the colony size the same. Contrary to this, many researchers [8, 53, 86] have reported an insignificant effect of colony size (ranging from 100-1200 µm) on the creep resistance in the stress range of 100-600 MPa (in general, equiaxed and duplex microstructures appear to be much more sensitive to grain size effects [3]). Es-Souini et al [31] suggest that a finer colony size might result in microstructural stability and reduced composite like behavior (more randomly oriented colonies are present). The presence of equiaxed γ grains on colony boundaries has been seen to degrade the creep resistance [8, 53]. No clear picture has emerged regarding the effect of colony boundary morphology on the creep strength. While some propose that presence of Widmanstätten laths enhances creep resistance [16], yet others report it as a deviation from a perfect FL structure and so a probable cause of higher creep rates [53]. Interlocking serrated colony boundaries have been reported to result in overall better creep resistance [8, 72]

c) **Microstructural evolution during creep**

For a fully lamellar structure to have satisfactory creep resistance, it needs to maintain sufficient microstructural stability during long term exposure at creep testing
temperatures (and envisaged component operating temperatures). However microstructural changes have been observed for the fully lamellar structure when exposed to high temperature for substantial periods of time [19]. It has been proposed that a fine lamellar structure with large volume fractions of $\alpha_2$, is far from equilibrium and is thus not a stable structure [27].

The FL alloys exhibit dissolution and thinning of $\alpha_2$ and coarsening of the gamma lamellae during stabilization treatments prior to and during creep [1, 14, 19, 30]. The coarsening process in long-term exposure experiments can lead to an almost total dissolution of the $\alpha_2$-phase in alloys with considerably lesser $\alpha_2$ (high Al content) [19]. Spheroidisation of the $\alpha_2$ laths has also been observed. The relative influence of stress and temperature is debated. While some claim that the spheroidisation of the $\alpha_2$ laths is essentially caused by high stresses [86], others observe similar microstructural changes in both crept samples and samples just exposed to high temperatures suggesting that temperature rather than stress is predominantly responsible [19]. Ripening of precipitates and formation of secondary $\gamma$ lamellae within $\alpha_2$ resulting in a packet like structure are among the other microstructural changes during creep [25]. The deterioration of the lamellar structure leads to a gradual increase in the strain rate (tertiary creep behavior) probably related to the gradual increase in the mean free path of the moving dislocations. Migration of interface boundaries has been suggested to sweep away dislocations that cause strain hardening in the primary creep regime, thus resulting in softening during the tertiary regime [86].
1.6. Modeling Creep in $\gamma$-TiAl alloys

The aim of any model is to approximate a physical process and create a mathematical representation that is capable of predicting aspects of the process within acceptable error. This is the aim of modeling creep behavior too. Accurate creep modeling helps reduce the total number of experiments and experimentation time. It also provides insight into the important creep controlling parameters and how creep properties can be improved. In this section, existing creep models for $\gamma$-TiAl will be reviewed and critiqued.

1.6.1. The "rule of mixtures" model [87]

This model suggested by Sobojeyo and Lederich uses the simple rule of mixtures, and constitutive equations for diffusional creep, dislocation glide and dislocation climb creep to calculate the strain rate of a composite FL structure. Several assumptions and estimations are necessary to provide values for all the parameters of the constitutive equations, but creep rates within an order of magnitude of those measured are obtained. Such a model predicts creep rate of lamellar alloys ranging between the creep rate for the constituent phases $\alpha_2$ and $\gamma$. However, it is now established that the creep rates of the lamellar structure is less than that of the constituent phases and failure to provide an explanation for this seems to be the major drawback of this model.

1.6.2. A modified Kelly and Street (KS) composite strengthening model [82]
This model, proposed by Bartholomeusz and Wert is a modification of the KS model for steady state creep of fiber reinforced composites. Correcting for the geometry of the lamellar structure and the random nature of individual grains, equations are developed for both the hard orientations. For lamellae parallel to the loading axis, isostrain between the $\gamma$ matrix and $\alpha_2$ reinforcement, and power law creep for the matrix is assumed. The compressive stress for strain rate is then given by the power law equation combined with the rule of mixtures, modified for the lamellar geometry. For the case of lamellae perpendicular to the loading axis, isostress condition is assumed. These two cases are envisioned to be the limits of predicted value of strain rate and a polycrystalline material with randomly oriented lamellae is expected to have properties between the two limits. The model has been seen to predict experimental values quite accurately.

Fiducial line techniques were used to show that interface sliding was minimal and that strain contribution comes completely from deformation in the individual phases. This model is in essence identical to the earlier "rule of mixtures" model. However it assumes (without proof) that the individual component phases work harden significantly more in the lamellar structure than as single-phase alloys. For this reason the resultant creep rate of lamellar structures is lower than that of the individual phases. There are several concerns with this model. Firstly, soft mode orientation deforms by a different mechanism and, it may be argued that the soft mode strain rate might fall outside that of the two hard modes. In view of the model's accurate predictions, it can be interpreted that the hard mode may be the creep rate-controlling in polycrystalline FL materials. The second concern is that no physical explanation is provided for the increased hardening of the individual phases in the lamellar microstructure. The physical meaning of the several
parameters used in the model are not addressed. It has been shown by Maruyama et al [88], that there is a strong effect of lamellar spacing and minimal effect of $\alpha_2$ volume fraction on the creep properties. This model does not explain the lamellar spacing effect and predicts a strong effect of $\alpha_2$ volume fraction. Though the experimental techniques appear valid, the model provides little insight into the physical process and comes across as a "fitting" exercise.

1.6.3. Micromechanical modeling [89]

A series of micromechanical models based on the "unit-cell" technique and employing finite element method (FEM) have been developed by Marketz et al, for predicting room temperature behavior. This has been modified suitably for predicting creep behavior. The model takes into account slip (of both ordinary and superdislocations) and twinning of both phases as the major deformation modes. Constitutive creep laws for both phases are employed. The constitutive law for the gamma phase takes into account the effect of lamellar spacing and lamellar orientation: these effects are refined from experiments done on PST crystals. Variously oriented colonies are considered for the FEM model. Compatibility between the lamellae and the grains is inherently assumed. The predictions for the polycrystalline FL alloys is in excellent agreement with experiment. The essence of this model it that it applies constitutive equations derived from PST alloys, to a polycrystalline material. The approach appears logical and justified. The concerns with the model in its current state are two-fold. The FEM model can only be as good as the constitutive equations that are used. The primary concern with this model is that like all the previous models, it provides
no insight into the mechanistics and the logic behind the choice of constitutive laws. For instance, the lamellar spacing effect in PST crystals may be vastly different from that in polycrystalline materials, which typically have smaller lamellar widths. Secondly, six grains each with 20 laths (2 of which are $\alpha_2$) is used for the 2-D FEM model. All laths are assumed to be of the same width. The authors themselves suggest that the size of the model may not be representative of the actual material. There is scope for large improvements in the model, as a more realistic picture of the lamellar microstructure is employed for the FEM model.

1.6.4. The Modified Jogged Screw Model [58]

Cusped screw dislocations have been often observed during high temperature deformation of $\gamma$-TiAl. The cusps have been identified as tall jogs. While several authors have tried to incorporate this observation into models for explaining the flow stress anomaly, Viswanathan et al. were the first to use this for modeling creep behavior in equiaxed $\gamma$-TiAl. The concept is that the motion of the screw dislocation is rate limited by the non-conservative motion of the jogs along the dislocation. The velocity of the screw dislocation assuming that jogs are all vacancy producing type is similar to one derived by Barrett and Nix [90], except that the jogs are no longer a Burger’s vector in height, but significantly taller. The other modification to this model has been made in the form of a stress dependent cap to height of jogs. Though this is a glide-controlled creep process, it predicts a stress exponent of 5 (which is typical of climb controlled processes). The predicted values of strain rates compare favorably with experimental values for two different temperatures. This model appears to be the first of its kind based on a thorough
analysis of the deformed microstructure. The drawbacks of this model are that several assumptions about the functional dependencies of model parameters are made without experimental evidence or satisfactory physical explanations. The model makes predictions only for equiaxed microstructures and not for the FL microstructures which are of greater engineering interest. This model will be thoroughly dissected and analyzed in Chapter 3. of this thesis.

1.6.5. "Interacting elements" model. [91]

Daehn et al. have tried to use a model involving interacting elements for predicting creep transients and the lamellar spacing effect in FL microstructures. This is based on a stochastic "cellular automaton" model, which involves a 2d array of elements. Each element is assigned a strength and when the local stress state exceeds this value, it slips, and simultaneous sheds load to its neighboring elements. The total strain is the average of strain produced in each element. The model behaves like an isostress model at early stages and as an isostrain model at longer times. Two types of obstacles are perceived: the first is related to jogs observed along the dislocation length and the second is related to penetration of the lamellar interface. The jogged-screw model developed by Viswanathan et al [58] has been used as the basis for modeling the first type of obstacles. The second type of obstacle is assumed to be stronger than jogs and the total number of these harder obstacles is proportional to the number of interfaces (and hence inversely proportional to the lamellar spacing). Both types of obstacles are overcome by thermal activation. Simulations based such a cellular automaton model is able to reproduce the primary transient behavior satisfactorily (power laws between strain and time are
predicted). It also predicts higher creep rates for the coarse lamellar specimens. The strengthening due to fine lamellar spacings is a direct result of having a higher density of harder obstacles. Though the obstacles are real, the actual strengthening mechanism of either of these obstacles is not considered (the authors acknowledge this). The emphasis has been on rationalizing the shape of the creep transient. It appears that the simulations would predict similar primary transients, in any system with two types of obstacles.

There are a few other models, which will not be reviewed in detail. These include a "diffusional creep" model [92] for predicting the low stress creep behavior of FL microstructures, a modified "continuum damage mechanism" model [93] that includes dislocation creep, grain boundary sliding and dynamic recrystallization, and "cavitation damage predicting FEM-based" model [94] that assume dislocation creep and grain boundary sliding as creep mechanisms.

1.7. Motivation and objectives of this research.

It is clear that creep literature of γ-tiAl is riddled with inconsistencies and uncertainties with respect to creep mechanisms. Interaction between the different microstructural and compositional variables masks and muddles the interpretation of results. Though broad trends are beginning to evolve, there is a need to confirm these trends on "controlled microstructures".

It is not possible to consider and account for all the microstructural and compositional variables, though it is definitely possible to evaluate which factors have the most significant effect on creep. In view of this, the aim of this research will be essentially focussed on evaluating the creep performance of FL alloys and on developing
a physically-based creep model. While the single phase gamma and two phase alloys in the equiaxed microstructure may not be of immediate engineering use, the understanding of creep mechanisms in gamma seems absolutely indispensable for the understanding of creep behavior in FL alloys. Chapter 3 is dedicated to the understanding of creep mechanisms in equiaxed γTiAl alloys. Emphasis will be on re-evaluating and revising the jogged-screw model. Chapter 4 is devoted to the determination of phenomenological and dislocation-level mechanistic aspects of creep deformation in FL alloys. The effect of lamellar spacing and orientation will be assessed. The combination of experimental and modeling investigations leads to the enrichment of one method by the other. So this chapter will concentrate on the development of a physically-based creep model in FL microstructures. Chapter 5 deals with the characterization of microstructural evolution during aging and creep. Possible damage and tertiary creep mechanisms are suggested. The role of microalloying is also assessed. Chapter 6 will provide a summary for this work and recommend future directions.
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Table 1.1. Properties of TiAl alloys compared with Ti$_3$Al alloys and superalloys [2]

Figure 1.1. A comparison of Young’s modulus values for various high temperature alloys [2]
Figure 1.2. The binary Ti-Al phase diagram [2]

Figure 1.3. The crystal structure of $\gamma$. The darker atoms are Ti atoms and the lighter ones are Al atoms.
Figure 1.4. The crystal structure of $\alpha_2$. The darker atoms are Ti atoms and the lighter ones are Al atoms.

Figure 1.5. The major types of microstructures for the $\gamma$-TiAl based alloys: equiaxed, duplex and fully lamellar [2].
Figure 1.6. Atom configuration of \{110\} planes showing the three types of interfaces in \(L1_0\) structure. (Small circles represent the atom positions on the plane one above or below the plane with large circles. Solid and open symbols refer to Ti and Al atoms.

Figure 1.7. A typical creep curve.
Figure 1.8. The ordinary and superdislocations are indicated for a L1₀ structure.

Figure 1.9. Normalized yield stress and work hardening rate at 1% plastic strain in Ti-51Al-2Mn polycrystals as a function of temperature (in °C). The yield stress anomaly is evident [59].
Figure 1.10. The orientations of PST crystals characterized by the angle $\phi$ subtended by the lamellar interfaces to the loading axis.

Figure 1.11. Anisotropy of room temperature yield strength and ductility in PST crystals is evident. [2]
Figure 1.12. A comparison of the creep behavior of single phase gamma, duplex and FL microstructures. The performance of FL alloys is superior to the other microstructures [2].

Figure 1.13. The minimum strain rate versus stress data for several alloys in the equiaxed microstructure is compared. The stress exponent is between 5 and 6 for all the alloys and for a range of temperatures.
Figure 1.14. The minimum strain rate versus stress data for several alloys in the fully lamellar is compared. The stress exponent is 2-3 at low stresses and is 8-10 at high stresses. The data is for one temperature. The strengthening effect of Cr and Nb at high stresses is evident.
CHAPTER 2. MATERIALS AND EXPERIMENTAL PROCEDURES

This chapter describes the different materials used in this study and also describes the mechanical testing procedures and characterization techniques that were most often used. Materials and experimental procedures, other than those described in this chapter, will be briefly described wherever appropriate.

2.1. Materials studied

Three different base alloys were chosen for this study. A simple binary gamma TiAl alloy was selected in the equiaxed and FL microstructure for studying the underlying creep mechanisms. A more complicated “K5” and “K5SC” alloy was picked for the study of microstructural effects (including strengthening mechanisms and microstructural stability) in FL microstructure. The nominal composition and the heat-treatment schedules for these materials, and the eventual microstructures obtained are described in detail below.

a) **Ti-48Al binary alloy**: The binary alloy has a nominal composition of Ti-47.86Al-0.116O-0.016N-0.041C-0.076H (in at.%). Cylindrical blanks of the forged alloy were heat treated at 1473 K in the( α + γ )two-phase region just above the eutectoid temperature and followed by a stabilization treatment at 1173 K for 6 hrs. This heat treatment yields a near-gamma microstructure with γ grains in the equiaxed
morphology (grain size in the range of 20-50 μm). The microstructure has a small volume fraction of lamellar grains. These samples will be referred to as Ti-48Al (EQ) henceforth. The typical microstructure is shown in Figure 2.1. Blanks of similar composition (Ti-48Al) were heat treated at 1658 K (above the α transus) to ensure a fully lamellar structure. These samples were also given a similar stabilization treatment. Different cooling rates were employed to get different lamellar spacings. These samples will be referred to as Ti-48Al (FL). The grain size (or “colony” size) is larger than 100 μm [1].

PST crystals of the binary Ti-48Al composition were prepared using a float zone technique in a Crystallox furnace using 10 mm diameter rods. This rod was then oriented using a Phillips XRG 3000 back-reflection Laue camera operating at 30 kV and 20 mA.

a) **K5 alloy:** The nominal composition of the K5 alloy is Ti-46.5Al-2.0Cr-3.0Nb-0.2W (in at. %). These alloys were isothermally forged to 90%. Blanks of K5 were given a heat treatment involving solution treatment (in the single-phase α phase field) at 1633 K for 5 minutes and furnace cooling to 1473 K, followed by air cooling to room temperature. The microstructure obtained thus is fully lamellar. Subsequently the blanks were aged (stabilization treatment) at 1173 K for 24 hrs in a tube furnace. All heat-treatments were performed in air. The grain size is 100-200 microns. An SEM image of the K5 alloy is indicated in Figure 2.2.

c) **K5SC alloy:** The K5SC alloy is in essence the same as the K5 alloy but has additional C and Si as interstitial elements and the nominal composition is Ti-46.0Al-1.8Cr-3.0Nb-0.2W-0.1C-0.2Si. The solution treatment given to this alloy is identical

54
to that for the K5 alloy. These alloys were typically aged similar to the K5 alloys (at 1173 K for 24 hrs). However the aging temperature and time was varied to study the effect of these on the microstructure and creep properties. These treatments will be discussed in chapter 5

2.2. Creep Testing

Cylindrical creep samples were prepared from the Ti-48Al (eq) and Ti-48Al (FL) alloy blanks and tested in tension at 768 and 815 °C. Both monotonic creep tests (tests under a constant load) and stress increment tests were performed. These tests were performed at University of Cincinnati and the details of these tests are described elsewhere [1].

Parallelepiped samples of dimensions shown in Figure 2.3 were machined from blanks of Ti-48Al (Eq), K5 and K5SC and PST alloys for compression creep testing. The PST samples were prepared in the N, A and B orientations such that the compression axis is perpendicular, parallel or at 45° to the lamellar interface, respectively. Care was taken to maintain the square faces of the sample (those in contact with the compression cage platens) parallel. These were creep tested in compression on an ATS Series 2410 lever-arm creep testing frame shown in Figure 2.4. An Inconel based compression cage equipped for "averaging" extensometry was used. All connectors in the load train are also made of Inconel.

The extensometer consists of a "rod in a tube" assembly. The rod is connected to the upper platen while the tube is connected to the lower platen of the compression cage.
An LVDT (Linear Variable Displacement Transducer) was used to measure the change in dimensions. The "mantle" of the LVDT is connected to the crosshead attached to the tube, while the "core" of the LVDT is connected to the crosshead attached to the rod. The LVDT thus senses relative motion between the platens. A load cell connected to the load train was used to measure the load applied. Signal conditioners provide excitation voltages to the load cell and the LVDT. They also amplify and provide low-pass filtering to the output signal. The LVDT signal was further cleaned for noise using another low-pass filter. Such filtering leads to a voltage accuracy of the LVDT signal down to +/- 2 mV which corresponds to a dimensional accuracy of +/- 1.2 microns. The load cell and the LVDT are zeroed and calibrated every few tests. The calibration curves are shown in Appendix 1.

The load cell signal and the LVDT signal were connected to an 8-channel NATIONAL INSTRUMENTS data acquisition board. This DAQ board was connected to a Macintosh computer running a LABVIEW program equipped to display and record the signals from the LVDT and the load cell (and a thermocouple, if need be). The rate of data acquisition can be controlled by the software and as many as 30 points can be collected in a second per channel. The fastest rate of acquisition used for our creep tests was 10 points a second during loading and during the initial stages of creep. The rate was reduced gradually as the strain rate dropped. The acquisition rate was never smaller than one in every five minutes.

An ATS three-zone vertical split-tube furnace was used for maintaining the creep temperature. The furnace was powered and controlled by an ATS furnace controller with independent three-zone control (Barber-Coleman Series 15 variable-current PID control
The furnace has the capability to maintain three different temperatures in the three zones. In our case, the same temperature was maintained in all three zones so as to maximize the isothermal zone within the furnace. A K-type thermocouple was kept in contact with the sample throughout the test. The temperature data was not acquired unless there was any temperature change during the test. The furnace controller is able to maintain temperature to the accuracy of +/-0.5 K over extended periods of time (even months). The thermocouple calibration is indicated in Appendix 1.

Prior to the test, the dimensions of the sample are accurately measured (to within a micron) and the load corresponding to the initial uniaxial applied stress is calculated. The weights required to achieve this load is tested on a dummy sample of similar dimensions. The applied load is accurate to within 1 lb which corresponds to an accuracy of less than 0.1% (for typical sample dimensions and an applied stress of around 300 MPa). The weights are unloaded using a screw-driven elevator and the dummy sample is exchanged for the actual sample. All the sliding parts of the compression cage are lubricated using powder-graphite. The sample is then heated under a small load (~30 lbs) so as to prevent the sample from getting misaligned during heating. The ends of the tube-furnace are insulated. The LVDT unit is fan-cooled and maintained at room temperature. Proper care was also taken to ensure free movement of the LVDT core inside the mantle. Under proper insulation the furnace heats up quite rapidly (10-15 degrees a minute) and reaches the target temperature within an hour. If needed, minor adjustments are made to the sample temperature. The sample is allowed to equilibrate at this temperature for 1 hr or till the signal obtained is stable for over a half-hour. The heating curve is shown in Appendix 1.
The loading/unloading system comprises of an elevator that is screw-driven by a motor. The weights sit on a pan resting on the elevator. While loading the elevator goes down and gradually loads up the sample. There is a sensor at the top of the elevator which senses the bottom of the pan and drives the elevator down so that it maintains a constant 1/2 inch distance from the pan. This also ensures that the sample is never unloaded when the pan moves down during the test. There are other proximity switches which prevent the elevator from moving too far up or down. There is also a safety switch which prevents the lever arm to come too close to the testing frame itself. Whenever these switches are tripped, the elevator stops being operational and coming down of the lever-arm and the pan is broken by the elevator. Typical motor actuated loading rates are 120-170 lbs/s, which corresponds to an initial strain rate of 2-6e-4 for a sample of standard dimensions.

All tests were conducted under constant load conditions. Several of these tests were monotonic tests, which involved a single load throughout the test. The first sample is let to creep until it acquires substantial strain and the subsequent samples are crept to pre-determined strain levels. The tests were performed for a range of stresses (100-400 MPa) and temperature (700-850°C). Stress jump and stress drop tests were also performed to obtain information about the stress exponent and the transient behavior. These tests were conducted by adding or removing load instantaneously while making sure that no shocks are imparted (thus minimizing the ringing). When the pre-determined strain level is achieved, the test is terminated by turning off and opening the furnace. This was done to rapidly cool the sample under load.
The load and displacement raw data was analysed, calibrations applied to, and expressed in terms of strain and time. All strain accumulated after the loading is complete is designated as the creep strain. The instantaneous plastic strain during loading is ignored. There is significant plastic strain accumulation during loading (in the range of 0.05% to 0.2%) and so the dynamic modulus is often quite different from the static modulus. This seems to be a major drawback of screw-driven loading systems, which inherently take a longer time to load than hydraulic systems. The screw-driven system has however has the considerable advantage of causing minimal ringing after loading.

The microstructures in the crept and uncrept samples were characterized using transmission electron microscopy.

2.3. Transmission Electron Microscopy.

The crept samples were sectioned normal and parallel to the loading axis using a diamond saw on Struers Accutom 50. These sections were 500-600 microns thick. These discs were then ground carefully using 400 and 600 grit paper down to 250 microns. 3mm discs are made out of these sections using a slurry drill. Low speed and load was employed and a SiC slurry was used. The direction of the loading axis with respect to the disc was noted. These 3mm discs were further ground down to about 170 microns using 800 and 1200 grit paper and again ensuring that the sample damage caused is minimum.

These foils were subsequently polished on either a Struers Tenupol-3 twin-jet electro-polisher or a Fischione submerged twin-jet electro-polisher. The electro-polishing solution consists of 65% ethanol, 30% butan-1-ol and 5% perchloric acid. After trial and
error, the ideal conditions were found to be at a voltage of 20V (current of \(~30\) mA) and temperature of \(-40^\circ\)C. Prior to TEM observation, all samples are plasma cleaned.

Observations on the microstructures were conducted on a Philips CM200 transmission electron microscope with a LaB\(_6\) filament, and operated at 200 kV. Conventional TEM techniques were employed. Two-beam imaging conditions were used for imaging dislocations. Standard g.b analysis was used for characterizing the dislocations. CBED patterns were used for measuring the sample thickness (used later for measuring the dislocation density). Compositional measurements were done by EDS on the TEM. For the lamellar alloys centered dark field images using \(\alpha_2\) reflections were used to image the \(\alpha_2\) laths. Similar techniques were used to image the different phases (B2, silicides and carbides). The measurements of the lath spacing were done by tilting the sample so as to get the lamellar interfaces edge-on. A line intercept method, involving measurement of lamellar widths along a line perpendicular to the lamellar interfaces, was used for measuring the lamellar thickness distribution. \(\alpha_2\)-lath spacing distributions were compiled. Volume fraction of \(\alpha_2\) and \(\gamma\) was extrapolated from the line-intercept method. The statistics shown in the following chapters was measured over several grains in several TEM samples.
LIST OF REFERENCES

1. G. B. Viswanathan, Microstructural effects on the tensile and creep behavior of Ti-48Al based gamma titanium aluminides, University of Cincinnati, Cincinnati (1997).
Figure 2.1. Optical micrograph of the Ti-48Al (EQ) alloy

Figure 2.2. SEM micrograph of the as-solutionized K5 FL alloy.
Samples were EDM'd and all six faces were ground flat to within within 0.05 mm. The top and bottom surfaces ensured to be parallel.

Figure 2.3. Dimensions of the compression testing sample
Figure 2.3. The lever arm creep testing frame
3. CREEP IN EQUIAXED γ-TIAL

3.1. Phenomenology of creep in equiaxed microstructures.

Typical creep curves for the equiaxed microstructure are shown in Figure 3.1 at different stress levels. The same curves are also shown in log strain-log time plots [Figure 3.2]. Several tests were conducted at one stress level to confirm the repeatability of these experiments. Figure 3.3 confirms consistency of creep results between different samples. Keeping this in mind other creep tests were not repeated.

The creep rate appears to exhaust with time, indicating an extended primary creep regime with little hint of tertiary creep. The minimum creep rate is plotted as a function of stress for two different temperatures in Figure 3.4. These values were obtained through a series of stress jump tests. Figure 3.4 shows that the creep response obeys a power-law equation relating the strain rate to the stress as

\[ \dot{\varepsilon} = A \cdot \sigma^n \cdot \exp\left(-\frac{Q}{RT}\right) \]  

(3.1)

where \( A \) is a constant, \( Q \) is the creep activation energy, \( R \) is the gas constant and \( T \), the temperature. It is seen that the equiaxed structures exhibit a stress exponent \( n \) between 5 and 6. Similar results have been widely reported [1-8]. [Figure 1.13]

Several monotonic creep tests were conducted at temperatures ranging from 760°C to 845°C. The typical effect of temperature is shown in Figure 3.5. The minimum creep rates are plotted as a function of 1/T on a log-log plot (for different strain levels) in
Figure 3.6. A closer look at equation (1.1) indicates that, the slope of such a plot is \(-Q/R\). Creep activation energy value \(Q\), can thus be extracted.

The effect of temperature on the creep rate is essentially two-fold. First, it affects the diffusion coefficient through an Arrhenius type equation. Second, an increase in temperature typically causes a drop in the shear modulus. This drop in modulus has to be accounted for through a correcting function such that true activation energy for creep is obtained. This is indicated by the equation:

\[
Q_c = Q + \frac{nRT^2}{E} \frac{dE}{dT}
\]  \hspace{1cm} (3.2)

where \(Q\) is the uncorrected activation energy (found from the slope of curve), \(n\) is the measured stress exponent, \(E\) is the elastic modulus and \(dE/dT\) is the change in modulus with temperature.

Figure 3.7 indicates the change in Young's modulus as a function of temperature. Other researchers have found a similar temperature dependence for the Young's modulus [9, 10]. The slope of this curve is used in equation (3.2). The correction factor to the activation energy varies in the ballpark of -22 to -28 kJ/mol in the temperature range of interest (for \(n = 6\)). An average value of this correction factor (-25kJ/mol) is picked and the "true" creep activation energy for creep \(Q_c\) is evaluated.

The value of the "true" creep activation energy has been plotted as a function of strain in Figure 3.8 and compared to activation energy for lattice self-diffusion \(Q_d\) (295kJ/mol), and the activation energy for pipe/grain boundary diffusion \(Q_p \sim Q_{gb}\), assumed equal to \(Q_d/2\) [4, 11, 12]. It is evident that the creep activation energy at steady state (~ 2% strain) compares favorably with that for lattice diffusion, indicating that the
creep mechanism at steady state in this temperature range and at 300 MPa is lattice diffusion. The dependence of activation energy on strain is not clear. The activation energy is seen to be quite large at large strains. This may be due to temperature-dependent substructural parameters (in the context of the jogged-screw model). On the contrary this may just be due to insufficient data. Typically when creep is controlled by pipe diffusion, the activation energy drops with increasing strain. Since this is not observed in this case, it provides support that creep mechanism is controlled by diffusion in the lattice, rather than by pipe diffusion.

3.2. Deformation microstructure

Sub-grain formation typically observed for a stress exponent of 5, is not seen in the minimum strain rate regime. Dislocation tangles and networks are seen only at large strains (above 4%). The dislocation density appears to be slightly higher near the grain boundaries than in the grain interiors. Figure 3.9 and 3.10 show typical deformed microstructures. At strains corresponding to the minimum creep rate, the deformation microstructure is dominated by 1/2[110]-type ordinary dislocations. The dislocations tend to be elongated in the screw orientation and appear to be frequently pinned along the length. The segments on either side of the pinning points are bowed-out forming cusps. The observation of dislocation debris in the vicinity of the pinning points is not uncommon [Figure 3.11]. Similar dislocation structures have been frequently reported to dominate creep microstructures [5-8, 13-16]. Figure 3.12 and 3.13 are high temperatures microstructures observed by other researchers that show considerable similarity to what we have observed.
In an attempt to characterize such pinning points (on samples deformed at a constant strain rate of $10^{-4}$/s), Sriram et al. have used the technique of analysing stereo-pairs of images tilted to known positive and negative angles about the general glide plane of the dislocation [13]. This technique of stereo-pair images has lead them to conclude that the bowed out segments almost exclusively lie on the parallel planes separated by several Burger's vectors. They identify the pinning points as jogs that are anywhere between 5 to 48nm in height. The stereo-pair technique has also previously been used by Viguier et al [5] but they conclude that the bowed out segments lie of different \{111\} planes. They do not quantify the jog heights, though they do measure the jog density (along the length of the dislocation). Viswanathan et al have employed similar stereo-pair imaging techniques on crept samples and have confirmed that these cusps are indeed associated with jogs [8]. They too indicate that the jogs are tall, often up to 40nm in height. The origin of tall jogs will be discussed in detail in the next section (Section 3.3).

The possibility that these cusps are related to extrinsic pinning points (oxide precipitates) has been previously suggested by Kad and Fraser [17] and Messerschmidt et al [18] However, the alloys used in their investigations are single phase \(\gamma\)-alloys. The actual oxygen content in these alloys exceeds the solubility limit (~300 at.ppm)[19]. It has been argued that the presence of \(\alpha_2\) as the second phase acts as a scavenger of oxygen and reduces the oxygen content in the \(\gamma\)-phase [20-22]. In the two-phase Ti-48Al alloy used in our investigation, residual oxygen levels in \(\gamma\) are assumed to be low enough to suppress precipitation of oxides. The lack of direct microscopic evidence of precipitates and the observation that the pinning point density increases with temperatures (not expected with precipitates) lends further support to our hypothesis.
3.3. Formation of tall jogs

A pure screw dislocation can slip on any plane, which contains it. When the screw dislocation moves from one glide plane to another, it is said to have cross-slipped. The section of screw dislocation that has cross-slipped can slip back onto the primary glide plane. This is termed as double cross-slip. Such a process leading to the formation of jogs as shown in Figure 3.14, appears to be related to the nature of the dislocation core. The screw dislocation in $\gamma$-TiAl has a very compact core, as suggested by simulations [23], weak beam studies [24] and HRTEM observations [25, 26]. This can be attributed to the very high CSF energy which is estimated to be in the range of 400-600mJ/mol [27-31].

The compact nature of the screw dislocations makes cross-slip events very easy.

Though the penchant of the screw dislocation for cross-slip, in $\gamma$-TiAl is well recognized, the mechanism by which tall jogs are created is still intriguing. There are two distinct scenarios possible: a tall jog may be created by repeated coalescence of unit jogs of the same sign or it could also form directly by a double cross-slip process involving significantly larger cross-slip step. We will try to reason out why one mechanism appears more probable than the other.

First let us consider the scenario where tall jogs are formed by the coalescence of unit jogs of the same sign. This necessarily requires that a large number of jogs are of the same type, so that their coalescence will lead to increase in the jog height rather than annihilation. If we assume that the unit jogs nucleate and collect at the tall jogs with time (due to conservative motion), any effective growth in jog height will occur, only if the unit jogs that nucleate (next to the tall jog) are mostly of the same type as the tall jog.
The compact nature of the screw dislocation core in \( \gamma \)-TiAl also suggests that a high Peierls stress exists, which makes its motion sluggish. Typically, the mobility of dislocations is inversely proportional to the width of the core [32]. Atomistic studies confirm that the screw orientation of the \( \frac{1}{2} \langle 110 \rangle \) dislocation has the greatest friction stress, suggesting that the motion of these dislocations would be rate limiting [5, 23, 25]. Electronic density calculations too suggest that deeper Peierls valleys are aligned along the \(<110\rangle\) directions [33]. The preponderance of dislocations oriented in the screw direction supplies microscopic evidence for this. In view of the low mobility, the movement of the screw dislocations occurs via kink nucleation and expansion. It has been shown by Rao et al, that it is relatively easy to expand kinks formed on the screw dislocation (the friction stress was measured to be about 30 Mpa) [34]. So it appears that the rate-controlling step would be the nucleation of kinks rather than expansion.

In light of this, if the screw dislocation throws out a kink in either the primary or the secondary slip plane, it is quite likely that the kinks expand rapidly ("unzipping" of the kink) and dislocation moves forward. If the kink is formed on the secondary slip plane, rapid kink expansion would cause the dislocation to move on the secondary slip plane before one of its sections cross-slips back onto the primary slip plane such that a jog is formed. (Figure 3.15). So, under such conditions jogs are not formed.

In a hypothetical case, we can assume the kink nucleation rate to be substantial and comparable to the rate of kink expansion. The interaction of two kinks lying on different \{111\} planes can give rise to the formation of unit jogs. Such a mechanism was proposed initially by Furubayashi et al [35], and was assumed to be operational in the case of \( \gamma \)-TiAl by Viguier et al [5]. The formation of the jog is this case is illustrated in
Figure 3.16. Even if numerous unit jog-pairs are present, this mechanism will lead to the formation of an equal number of both types of jogs (and frequently alternating in sign). Any conservative motion and jog-jog interaction will only lead to jog annihilation events and the jog height will not increase (Figure 3.17). **Note:** The above mechanism for jog coalescence presumes that kink nucleation rates are high, and this in itself appears to be an invalid assumption. Also note that this mechanism *could* lead to the formation of tall jogs if the kinks thrown out are several jumps over the Peierl's valley (instead of unit kinks) and if there exists a spectrum of macrokink jump length. Such a situation is feasible under Caillard's "locking-unlocking" model, whereby the kink can cross several Peierls valleys before getting locked again [55].

Actual TEM observations made on samples crept for very small times indicate that jogs are already quite tall. All this seems to suggest that formation of tall jogs purely because of coalescence events of unit jogs is statistically less probable. This also seems to indicate that tall jogs are probably created directly by a double cross-slip process involving significantly larger cross-slip step. Now let us analyze this other possible mode for the formation of tall jogs.

At temperatures corresponding to creep deformation, cross-slip can be purely thermally activated. In the presence of the applied stress, cross-slip will be biased such that the applied stress does work. This results in a great majority of cross-slip events taking place in the forward direction. If the probability of cross-slip on the secondary slip system is $P$, the probability that the cross-slipped segment moves at least $n$ atomic steps in the secondary slip plane before cross slipping back to the primary plane is $P^n$. So the probability of creation of tall jogs drops exponentially. In the absence of all inertial
effects, a majority of jogs, thus created, are unit or just a few Burgers vector tall. Any increase in jog height in this case can be accomplished only by jog coalescence events which cannot lead to the formation of tall jogs (since we have jogs of both signs) [Figure 3.17]. So, a purely thermally activated double cross-slip process cannot lead to the formation of tall jogs either.

In the absence of thermal activation, the moving dislocation will always travel on the most highly stressed plane. A screw dislocation will cross-slip only when it encounters an obstacle [32]. In the vicinity of an obstacle (could be due to stress fields from other dislocations), forward slip on the primary glide plane becomes impossible and cross-slip on to the secondary slip system is relatively favored. Once the obstacle is avoided, slip on the primary slip system would be favored yet again and would cause double cross slip [Figure 3.18]. Depending on the nature of the obstacle, this mechanism can lead to the formation of tall jog instantaneously. Double cross-slip is typically expected in samples in which the Schmid factor on the primary slip plane exceeds that on the cross slip plane [13]. This mechanism can lead to both forward and backward cross-slip as shown in Figure 3.18. In such a scenario, jog-coalescence events will lead to the increase in jog height. The kinetics of kink nucleation and migration will not be important here because the jog formation itself is controlled by the variations in the internal stress fields. This mechanism of double cross-slip as a local recovery process appears to explain the observation of tall jogs. The above argument did not take into account any inertial effects. These effects will only help in promoting the formation of tall jogs.
Though it is not completely clear whether tall jogs form by interactions between macrokinks ("locking-unlocking" mechanism) or by double cross-slip, the latter mechanism driven by internal stresses that make cross slip on the secondary slip plane favorable locally, appears more feasible. These internal stresses could be due to other dislocations (other screw dislocations), or even due to the dislocation debris. In the following section we will build the basis for a creep model based on the observation of tall jogs as pinning points.

3.4. Jogged-screw model.

Significant work was done in the late 50s and 60s towards the development of the jogged-screw model. The earliest suggestion of this mechanism came in 1956 from N. F. Mott [36]. He formulated a creep model based on the movement of jogged screw dislocations. Johnston and Gilman [37], in their seminal work, provided evidence for the presence of jogs on screw dislocations in lithium flouride crystals, using etch pit techniques. Hirsch and Warrington [38] and later Raymond and Dorn [39] developed creep models not unlike the one postulated by Mott.

Barrett and Nix [40] furthered the model, considering a number of variations including positive and negative jogs and accounting for pipe diffusion. They concluded that a majority of jogs cannot be vacancy absorbing, since that produces a stress dependence that is not observed experimentally. They assumed the velocity of a screw dislocation with equal numbers of positive and negative jogs to be the average velocity of these two jog types. This work was strongly criticized by Weertman in a series of letters and papers [41, 42]. His objection is based on a theorem by Cottrell, that during multiple
slip all jogs created must be vacancy absorbing ones to ensure mass conservation. In such a case there is a need for vacancy sources such as edge dislocations. He concluded that the motion of jogged-screw dislocations is thus controlled by the climb of edge dislocations and that these are associated processes. Nix, in his defense argued that jogs are not formed by intersection of two sets of mobile dislocation, but rather by the intersection of a mobile dislocation with immobile forest dislocations [43]. This circumvents the constraints imposed by Cottrell's theorem. Additionally, in this paper he reconstructed the model for equal numbers of positive and negative jogs and instead of using the "average" velocity, he partitioned the applied stress such that both types of jogs move at the same velocity. This model unlike the earlier model predicts a stress dependence that decreases with increasing stress (not observed experimentally). On the basis of this, he rejected the case where equal numbers of vacancy producing and vacancy absorbing jogs are present. He also suggested that for obtaining the proper stress dependence, screw dislocations must have an overwhelmingly large fraction of vacancy producing jogs.

3.4.1. Basis of the model for equiaxed γ-TiAl

The observation of cusped screw segments associated with jogs and the general absence of subgrains suggests that creep rate may be controlled by the motion of jogs that lie on the screw dislocations [8]. The models discussed above, assumed that jogs form by the interaction of dislocations moving on different slip systems and, thus assumed to be unit jogs. In our case, jogs are observed even when there is little activity on secondary slip systems (in a particular grain), confirming that jogs have not arisen due to dislocation
intersections. Our belief is that jogs are formed on screw segments by double cross-slip (and so both types of jogs are present). Subsequent motion of the screw dislocation then requires the non-conservative dragging of these jogs. This forms the basis of our creep model.

The jog does not lie on the primary slip plane of the screw dislocation, and so its motion (in the direction in which the screw dislocation moves) can only occur non-conservatively. Such a motion requires that the jog, emit or absorb point defects (vacancies and interstitials). The nature of the point defects and whether they will be absorbed or emitted depends on the sign of the jog.

A vacancy-emitting jog on a screw dislocation thus emits vacancies and moves along with the rest of the dislocation. Sustained motion of the screw dislocation will give rise to a super-saturation of vacancies behind the jog. This super-saturation depends on the rate at which vacancies are produced (and so the rate at which the jog moves forward) and the rate at which vacancies diffuse away. Super-saturation of vacancies makes it difficult for the jog to move forward and generate any more vacancies. This retarding force on the jog can be considered to be a chemical force and is the result of the super-saturation.

An oppositely signed jog can move forward by either emitting interstitials or by absorbing jogs. It has been shown that it is energetically more favorable for such a jog to absorb vacancies rather than emit interstitials [44]. Hence, for this case, vacancy under-saturation is created behind the jog, the value of which depends on the rate of vacancy absorption (jog velocity) and diffusion. This under-saturation once again creates a retarding force on the jog.
Given the concentration gradient in an ideal solution, the chemical force can be computed as the negative derivative of the chemical potential [40, 44]. The osmotic force per unit length of the dislocation is given by

$$F_p = \frac{kTb}{\Omega} \ln \frac{C_p}{C_0}$$

for vacancy producing jog (3.3)

$$F_a = \frac{kTb}{\Omega} \ln \frac{C_a}{C_0}$$

for vacancy absorbing jog (3.4)

where $k$ is the Boltzmann's constant, $T$ is the temperature, $b$ the Burger's vector, $\Omega$ is the atomic volume, $C_0$ is the equilibrium vacancy concentration, $C_p$ is the super-saturated vacancy concentration and $C_a$ is the under-saturated vacancy concentration. The total retarding force on a jog of height $h$ is given by:

$$f_p = \frac{kTbh}{\Omega} \ln \frac{C_p}{C_0}$$

for vacancy producing jog (3.5)

$$f_a = \frac{kTbh}{\Omega} \ln \frac{C_a}{C_0}$$

for vacancy absorbing jog (3.6)

In the absence of lattice friction forces, steady state is achieved when the retarding chemical force is equaled by the driving force (due to the applied stress). Or in other words, steady-state dislocation velocity is achieved when the net force on the dislocation line is zero. For a jogged screw dislocation moving self similarly as shown in Figure 3.19, this condition is given by

$$\tau \cdot b \cdot l = f_p$$

for a vacancy producing jog (3.7)

$$\tau \cdot b \cdot l = f_a$$

for a vacancy absorbing jog (3.8)

where $\tau$ is the applied shear stress and $l$ is the jog spacing.
By using equations (3.5), (3.6), (3.7) and (3.8), we can come up with the following equalities:

\[
\frac{C_e}{C_0} = \exp\left(\frac{\tau l \Omega}{k T h}\right) \quad \text{for a vacancy producing jog} \quad (3.9)
\]

\[
\frac{C_a}{C_a} = \exp\left(\frac{\tau l \Omega}{k Th}\right) \quad \text{for a vacancy absorbing jog} \quad (3.10)
\]

It is evident that for a given temperature, the retarding force depends on the super-saturated or under-saturated vacancy concentration behind the jog. Since the ultimate aim of this exercise is to evaluate the jog velocity, it is important to develop a technique for evaluating the super-saturated (or under-saturated) vacancy concentrations and in particular, the relation between these values and the jog velocity. To this effect, we have adapted Rosenthal's analysis of point, linear and plane sources of heat [45]. The following section delves deeper into Rosenthal's analysis where we exploit the fact that jogs can be treated as sources (or sinks) of vacancies.

### 3.4.2. Rosenthal's Analysis

The issue of a moving point or line source of vacancies in an infinite homogeneous medium is very similar to the problem of a moving point/line source of heat. The theory of moving sources of heat has been of significant interest to the welding engineer. Rosenthal developed the fundamentals of this theory and derived appropriate solutions for linear, two- and three-dimensional flow of heat in homogeneous media and specifically the movement of point, linear and plane sources of heat [45]. In this section, the development of the jogged-screw model by Barret and Nix [40], on the basis of
Rosenthal's analysis of moving heat sources is reviewed. The analysis is then extended to the case of tall jogs.

The differential equation for heat flow expressed in rectangular co-ordinates with reference to a fixed origin in a solid is given by:

$$\frac{\partial T}{\partial t} = \frac{1}{2 \cdot \lambda} \left[ \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right]$$  \hspace{1cm} (3.11)

where $T$ is the temperature; $x$, $y$, and $z$ are Cartesian co-ordinates, $1/2\lambda$ is the thermal diffusivity of the metal.

We now assume the source of heat to be moving at a constant speed of $v$ along the x-axis. There are two exactly identical ways of looking at this situation. The first, is to assume that the origin is stationary, while the source moves. The second is to assume that the origin itself moves at velocity $v$. This is a non-accelerating frame of reference and is thus identical to the first scenario. The problem can be further simplified by assuming that the origin "sits" at the source. Thus "$x$" in Equation (3.11) can be replaced by "$\xi = x - vt$", where $\xi$ is the distance of the point considered from the origin (and the source). Equation (3.11) can be rewritten thus:

$$\frac{\partial T}{\partial \tau} - v \frac{\partial T}{\partial \xi} = \frac{1}{2 \cdot \lambda} \left[ \frac{\partial^2 T}{\partial \xi^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right]$$  \hspace{1cm} (3.12)

It has also been shown [45] that if the source of heat is much smaller than dimensions of the solid, the temperature distribution around the heat source becomes constant. Or in other words, an observer stationed at the heat source does not notice any change in the temperature in the vicinity of the source, as the source moves along. This state in heat-flow is called quasi-stationary, and is mathematically expressed thus:
\[ \frac{\partial T}{\partial t} = 0 \]  \hspace{1cm} (3.13)

Thus Equation (3.2) can be rewritten as
\[ -v \cdot \frac{\partial T}{\partial \xi} = \frac{1}{2} \cdot \lambda \left[ \frac{\partial^2 T}{\partial \xi^2} + \frac{\partial^2 T}{\partial \eta^2} + \frac{\partial^2 T}{\partial \zeta^2} \right] \]  \hspace{1cm} (3.14)

This can be further simplified by making the following substitution:
\[ T - T_0 = \Delta T = e^{-\lambda \cdot \xi} \cdot \varphi(\xi, x, y) \]  \hspace{1cm} (3.15)

where \( T_0 \) is the initial temperature of the solid and \( \varphi \) is a function to be determined. This substitution leads to a simplified version of Equation (3.14).
\[ (\lambda \cdot v)^2 \cdot \varphi = \frac{\partial^2 \varphi}{\partial \xi^2} + \frac{\partial^2 \varphi}{\partial \eta^2} + \frac{\partial^2 \varphi}{\partial \zeta^2} \]  \hspace{1cm} (3.16)

This can be rewritten symbolically thus:
\[ (\lambda \cdot v)^2 \cdot \varphi = \nabla^2 \varphi \]  \hspace{1cm} (3.17)

This equation can be used to provide solutions for heat transfer problems in infinite and semi-infinite solids involving moving sources of heat.

Let us first consider the case of a point source of heat moving in a homogeneous solid medium. This involves the three-dimensional flow of heat from the heat. The boundary conditions for this case are the following:
\[ \frac{\partial T}{\partial \xi} \rightarrow 0 \hspace{0.5cm} \text{for} \hspace{0.5cm} \xi \rightarrow \pm \infty \]  \hspace{1cm} (3.18a)
\[ \frac{\partial T}{\partial \eta} \rightarrow 0 \hspace{0.5cm} \text{for} \hspace{0.5cm} \eta \rightarrow \pm \infty \]  \hspace{1cm} (3.18b)
\[ \frac{\partial T}{\partial \zeta} \rightarrow 0 \hspace{0.5cm} \text{for} \hspace{0.5cm} \zeta \rightarrow \pm \infty \]  \hspace{1cm} (3.18c)
and by considering a spherical surface $4\pi R^2$ drawn around the heat source with radius $R = \sqrt{x^2 + y^2 + z^2}$, we can represent the other boundary condition at the source:

$$-\frac{\partial T}{\partial R} 4\pi R^2 k \rightarrow q \quad \text{for} \quad R \rightarrow 0$$

(3.19)

where $k$ is the heat conductivity of the solid and $q$ is the rate of heat input.

By solving Equation (3.17) (in cylindrical co-ordinates), and applying the above boundary conditions we can come up with the following solution for $\varphi$

$$\varphi = Ce^{-\lambda R}/R$$

(3.20)

The corresponding temperature distribution is:

$$T - T_0 = \frac{q}{4\pi k R} e^{-\lambda R(x+y)}$$

(3.21)

### 3.4.3 A unit jog as a source of vacancies

First, let us consider the case of a unit positive or negative jog that acts as either a point source or point sink of vacancies. This situation was treated in detail by Barrett and Nix in their development of creep model involving the glide of jogged screw dislocations [40]. The diffusion of heat and vacancies in a homogeneous solid medium are similar problems. Many practical cases of heat flow can be directly used in diffusion problems by simply replacing $T$, the temperature with $C$, the concentration [46]. This similarity between the temperature profile around a moving source of heat and the concentration profile of vacancies around a moving source of vacancies (jogs) was identified by Barrett and Nix [40].
Using Rosenthal's Analysis and Equation (3.21), it is possible to express the super-saturation around a unit jog that "produces" vacancies as it moves:

\[
C_p - C_0 = \frac{v}{4\pi D_v b R} e^{-\frac{v(\xi + R)}{2D_v}}
\] (3.22)

This equation was obtained by performing the following substitutions:

1. \(T\), the temperature is replaced by \(C\), the concentration.
2. \(k\), the thermal conductivity and \(1/\lambda\), the thermal diffusivity of the metal are replaced by \(D_v\), the lattice diffusivity of vacancies.
3. \(q\), the rate of heat input is replaced by \(v/b\), the number of vacancies produced per second. As the jog moves forward by one Burgers vector \(b\), it produces one vacancy.

If the jog moves \(v\) meters in a second, then it has to produce \(v/b\) number of vacancies in that second. Hence this substitution.

The computation of the chemical force on the moving jogs, involves the calculation of the vacancy super-saturation (in the case of a vacancy-producing jog), just behind the jog. At this position \(\xi = -b\) and \(R = |\xi| = b\). Carrying out these substitutions in equation (3.22), we get:

\[
\Delta C_p^w = C_p - C_0 = \frac{v}{4\pi D_v b^2}
\] (3.23a)

Similarly, for the case of vacancy absorbing jogs, the vacancy sub-saturation right behind the moving jog is given by

\[
\Delta C_a^w = C_a - C_0 = -\frac{v}{4\pi D_v b^2}
\] (3.23b)
Here $C_p$ is the vacancy concentration behind a vacancy producing jog, $C_a$ is the vacancy concentration behind a vacancy absorbing jog, $C_0$ is the equilibrium vacancy concentration. $\Delta C_p^u$ is defined as the super-saturation behind a unit vacancy-producing jog and $\Delta C_a^u$ is defined as the under-saturation behind a unit vacancy-absorbing jog.

### 3.4.4 A tall jog as a source of vacancies

As reported in section 3.2, jogs in $\gamma$-TiAl are several hundred Burgers vectors in height. We thus require a scheme to calculate the vacancy super-saturation or under-saturation behind a tall jog. The solution to equation (3.17) was not provided by Rosenthal for the case of a finite length source of heat (the "heat-equivalent" of a tall jog).

A first order approximation was made by Viswanathan et al [8], by assuming the tall jog to be a Concentrated Moving Point (CMP) source of vacancies. In essence, if there is a tall vacancy producing jog that is $n$ Burgers vectors tall, the CMP approximation assumes that unit progress of the jog leads to the creation of $n$ vacancies. This assumption implies that the vacancy super-saturation behind the jog scales linearly with jog height, as shown below.

$$\Delta C_{p,CMP}^f = \Delta C_p^u \cdot n = \Delta C_p^u \cdot \left( \frac{h}{b} \right) \quad (3.24)$$

where $\Delta C_{p,CMP}^f$ is the super-saturation behind the tall jog under the CMP assumption, $\Delta C_p^u$ is the super-saturation behind a unit jog, $h$ is the height of the tall jog and $b$ is the Burgers vector. Super-saturation behind the unit jog was derived in the last section using Rosenthal's analysis (Eq (3.23a)).
A more appropriate model for a tall vacancy-producing jog is to assume that such a jog is a Finite Length Moving Linear (FLML) source of vacancies. The aim of the following exercise is to find out how well the CMP solution approximates the FLML solution. As in the last section, the following treatment will be for a vacancy-producing jog. The development is identical for a vacancy-absorbing jog and only the final result will be presented.

As mentioned earlier the solution to Eq (3.17) was not provided by Rosenthal for a FLML source [45]. However, we could take advantage of the principle of superposition for calculating the vacancy super-saturation. The tall jog can be envisioned to be a string of $n$ point sources. The vacancy concentration behind the jog is thus the superposition of vacancy concentration due to this linear string of $n$ point sources. This is shown schematically in Figure 3.20.

This can be mathematically calculated at each point behind the jog (for example at point O, right behind the jog in Figure 3.20):

$$
\Delta C_{p,FLML} = \int_{P_1}^{P_n} \Delta C_p(R).dR
$$

(3.25)

where $\Delta C_{p,FLML}$ is the super-saturation behind the tall vacancy-producing jog (at say, point O) under the FLML assumption. The integration is carried out within the limits of $P_1$ and $P_n$, which correspond to the two ends of the jog. Since the vacancy concentration profile around a point source is radially symmetric, the vacancy super-saturation at point O, due to any point source (say, $P_x$) depends only on the distance between O and that point. $\Delta C_p(R)$ is the super-saturation due to such a point source, which is a distance $R$. 
away from point O. This is mathematically identical to Eq (3.22) and is expressed below again:

\[
\Delta C_p(R) = C_p - C_0 = \frac{v}{4\pi D_b R} e^{-\frac{v(R + \xi)}{2D_b}}
\]  

(3.22)

The integration over the length of the jog is equivalent to superposing the vacancy concentrations.

Symbolic integration of the above integrand leads to an exponential integral (Ei) function, the evaluation of which is not simple. It is also important to note that the super-saturation is caused due to creation of vacancies at discrete atomic positions. Since this integration assumes the value of \( R \) to be continuous, we are bound to end up with erroneous results. However, the equivalent summation of Equation (3.25) at these discrete values of \( R \) is completely valid and is not complicated. This is the method adopted to achieve the vacancy super-saturation at a particular point. If the tall jog is \( n \) Burger's vector tall, the equation used is shown:

\[
\Delta C_p,FLML = \sum_{n=P_f}^{P_n} \Delta C_p(n)
\]  

(3.26)

The function \( \Delta C_p(n) \) is evaluated at O due to \( n \) points from \( P_f \) and \( P_n \), by calculating \( R \) at each point and substituting it into equation (3.25). This summation is then carried out at all points around the jog.

The vacancy concentration profile achieved under the FLML assumption is shown in Figure 3.21. This is compared to the vacancy concentration profile under the CMP assumption. This can be easily calculated by using equation (3.22) and additionally substituting \( v \) by \( n.v \), to account for the concentrated point source. \( n \) as defined earlier, is
the height of the tall jog in number of Burger's vectors. The tall jog in Figure 3.21 was assumed to be 20 Burger's vector tall.

It is evident that the vacancy concentration profile around the jog is significantly different under the two assumptions. The concentration gradient appears shallower under the FLML assumption. The tall jogs observed in the TEM are several hundred Burger's vector tall (more than an order of magnitude taller than in Figure 3.21). For such realistic jog heights, the difference in concentration profiles can be expected to be even more drastic. We can also compare the actual value of super-saturation at point O, as a function of jog height under the CMP and FLML assumptions. This is indicated in Figure 3.22. Also shown in the figure is the vacancy super-saturation behind an infinitely tall jog, which would be the limiting case.

This value was calculated again using Rosenthal's analysis for an Infinitely Long Moving Line (ILML) source of vacancies [45]. Again, the solution was derived for the two-dimensional flow of heat and will be adapted for the flow of vacancies. Since this situation essentially involves a two-dimensional flow (there is no flow in the z direction along which the source lies), Equation (3.4) can be rewritten, thus:

\[
(\lambda \cdot v)^2 \cdot \varphi = \frac{\partial^2 \varphi}{\partial x^2} + \frac{\partial^2 \varphi}{\partial y^2}
\]  

(3.27)

Assuming boundary conditions similar to the three-dimensional case (at infinity and at the source) and again using cylindrical co-ordinates, we can solve for \( \varphi \). The solution for the two-dimensional heat flow case, therefore is

\[
T - T_0 = \frac{q}{2\pi k} e^{-\lambda R} K_0(\lambda v R)
\]

(3.28)
This solution involves a modified Bessel function of the second kind and zero order, and is represented by the symbol \( K_0(\lambda v R) \). \( R \) is again the radius and is now defined as \( R = \sqrt{x^2 + y^2} \) and \( q' \) is the rate of heat of a linear source (per unit length). All other terms in Eq (3.28), hold the same meaning.

This solution can now be rewritten for the two-dimensional flow of vacancies instead of heat. We can make the same substitutions as in Section 4.4.3 for the terms in equation (3.28). \( q' \) is substituted by \( \nu/b^2 \). We get the following equation by doing so:

\[
\Delta C_{p,\text{ILML}}^{\text{i}} \equiv C_p - C_0 = \frac{\nu}{2\pi D_v b^2} e^{\frac{-\xi}{b^2}} K_0\left(\frac{\nu R}{2D_v}\right)
\]  

(3.29)

Here \( \Delta C_{p,\text{ILML}}^{\text{i}} \) is the super-saturation behind the infinitely tall vacancy-producing jog. We can thus calculate the super-saturation right behind the jog at \( \xi = -b \) and \( R = |\xi| = b \).

\[
\Delta C_{p,\text{ILML}}^{\text{i}} \equiv C_p - C_0 = \frac{\nu b}{2\pi D_v b^2} e^{\frac{-\xi}{b^2}} K_0\left(\frac{\nu b}{2D_v}\right)
\]  

(3.30)

This was the equation used to calculate the vacancy super-saturation behind an infinitely tall jog in Figure 3.22, which would be the limiting case.

The following observations and conclusions can be made from Figure 3.22.

1) It is clearly seen that the under the CMP assumption, super-saturation increases linearly with height of jog, while under the FLML assumption it increases almost linearly at short jog heights, but asymptotically tends towards the limiting super-saturation under the ILML assumption.

2) This asymptotic increase under the FLML assumption towards the limiting case (at large jog heights) indicates that FLML assumption provides a more realistic picture of the vacancy concentration environment around the jog. It is clear that at large jog...
heights, the super-saturation under the CMP assumption overshoots the limiting
super-saturation value by orders of magnitude and is clearly not an accurate
description of the tall jog.

3) It is thus, concluded that the CMP assumption previously made [8] to describe a tall
jog over-estimates the super-saturation behind the jog and makes dragging of such a
jog much more difficult. In reality, the super-saturation is much less (under the FLML
assumption) and so taller jogs drag more easily than is anticipated by the previous
CMP assumption.

4) The super-saturation predicted by the FLML assumption can be related to the jog
height though a correcting function $\delta(h)$ as shown below:

$$\Delta C_p' \equiv C_p - C_0 = \frac{v_c \delta(h)}{4 \pi D_1 b^2}$$

(3.31)

The correcting function is in essence the ratio between the super-saturation behind a
tall jog and a unit jog as a function of jog height. This ratio is expected to a
logarithmic function of the jog height. The exponential integral function (the result of
the integration) can be approximated to logarithmic function for very small values of
$v_c b/2D$. This value is indeed the case due to the small jog velocity (typically in the
range of $10^{-10}$ m/s). Since the functional form of the integration and the summation
will be essentially the same, a logarithmic fit was adopted to obtain the correcting
function. The form of the correcting function is shown:

$$\delta(h) = 0.17 + 4.528 \cdot \ln\left(\frac{h}{b}\right)$$

(3.32)

This correcting function will be subsequently used in Section 3.5.
5) The ratio between super-saturation behind an infinitely tall jog and a unit jog can be easily calculated using equations (3.30) and equation (3.13). This ratio is given below:

\[
\frac{\Delta C_{p,ILML}^i}{\Delta C_a^u} = 2 e^{\frac{v}{2D_v}} K_0 \left( \frac{vb}{2D_v} \right)
\]

(3.33)

As mentioned earlier \( \frac{vb}{2D_v} \) is an extremely small number and so \( \exp(\frac{vb}{2D_v}) \) in equation (3.33) is nearly equal to 1. The above ratio can be approximated thus:

\[
\frac{\Delta C_{p,ILML}^i}{\Delta C_a^u} = 2 K_0 \left( \frac{vb}{2D_v} \right)
\]

(3.34)

6) It is important to note that all the above calculations were made at 815°C, for a jog velocity \( v \) of \( 10^{-10} \) m/s. The lattice diffusivity for vacancies \( D_v \) was calculated from the lattice self-diffusion coefficient and the vacancy concentration thus:

\[
D_v = \frac{D_s}{x_0}
\]

(3.35)

\( D_s \) is the lattice self-diffusion coefficient and \( x_0 \) is the vacancy density (atomic fraction) at that temperature. The value of \( D_s \) was picked from reference [11] and the value of \( x_0 \) was picked as \( 10^{-8} \) [47]. It thus becomes very important to evaluate the sensitivity of the above analysis and especially the correcting function \( \delta(h) \) to the choice of these numbers.

First, we will look at the sensitivity of the correcting function to jog velocity. Using the Orowan equation (Equation (1.4)), it is possible to evaluate the actual velocity of the jog (the requisite dislocation density information was obtained, as will be shown in section 3.5.1). The jog velocity is typically seen to vary from \( 10^{-10} \) m/s to \( 10^{-7} \) m/s.
The correcting function has been evaluated for different jog velocities. This is indicated in Figure 3.23. It is obvious that the correcting function is almost totally independent of the jog velocity (from $10^{-15}$ to $10^6$ m/s). Even for an unrealistic jog velocity of $10^{-2}$ m/s, the correcting function is not significantly different from that for more realistic jog velocities. Hence, it is concluded that the correcting function is insensitive to the jog velocity. [Note: The integration of Eq (3.25), the approximation that $vb/2D_v$ is small and the resultant approximation of the exponential integration function to a logarithmic function actually predicts that the correcting function would be totally independent of the jog velocity for realistic tall jog heights].

Second, let us explore the sensitivity of the correcting function to lattice diffusion coefficient for vacancies $D_v$. Figure 3.24 shows the correcting function for a range of $D_v$ values. It is evident once again that the correcting function is insensitive to the choice of the value for $D_v$. This too can be proved analytically, by making approximations as in the last case.

What seems to be the crucial parameter is the actual ratio between $v$ and $D_v$. This ratio figures prominently in many of the previous equations. The sensitivity of the correcting function to the $v/2D_v$ is shown in Figure 3.25. The conclusion once again is that the correcting function itself, seems to be very rigid and insensitive to the choice of values, for realistic tall jog heights (hundreds of nanometers).

The change in the ratio between $v$ and $D_v$ does however affect the super-saturation limit for an infinitely tall jog. The ratio between super-saturation behind an infinitely tall jog and a unit jog is given by equation (3.33). The dependence of this ratio on
$v/2D_v$ is shown in Figure 3.26. The dependence is however very weak: orders of magnitude change in $v/2D_v$ causes a very small change in the limiting ratio.

The conclusion that can be drawn from Figures 3.23 through 3.26 is that the correcting function is highly insensitive to the choice of $v$ and $D_v$ for realistic jog heights. Sensitivity to the values of $v$ and $D_v$ is small even for infinitely tall jogs. For all practical purposes, the correcting function is solely a function of the jog height.

In the next section we will try to briefly analyze the validity of Rosenthal's analysis.

### 3.4.5. Validity of Rosenthal's Analysis

This section addresses the assumptions made in the above treatment. The validity of these assumptions and possible shortcomings are discussed.

1. While positive jogs are vacancy producing, the negative ones can produce interstitials or absorb vacancies. It has been shown by Friedel [48], that it is energetically favorable for such a jog to absorb vacancies rather than emit interstitials. Interstitials usually have significantly larger formation energies than vacancies (typically by a factor of 5, for common metals) [44, 48]. The thermal concentration of interstitials is thus negligible compared to that of vacancies at all temperatures below the melting point. Since vacancies are the majority point defect in self-diffusion, it is fair to assume that the jogs move by absorbing vacancies, rather than by creating interstitials [44].
2. The Rosenthal's solution is based on a quasi-stationary situation. This assumption appears to be valid, since the jog height is much smaller than the dimensions of the solid, and consequently the vacancy distribution around the jog becomes constant.

1. The theory for evaluating the jog velocity is based on a quasi-equilibrium assumption. This means that local vacancy equilibrium is assumed at the jogs. This in essence implies that the rate controlling mechanism is the mobility of vacancies away from the jog rather than the creation of vacancies (for a vacancy-producing jog). This ensures that the local vacancy concentration is in equilibrium with the jog. If the creation of a vacancy involves a significant energy barrier, this equilibrium concentration would not be reached and this step would be rate controlling. In such a case, the quasi-equilibrium approach is not applicable. Such barriers have been proposed to be likely only in metals with low stacking fault energy and thus widely extended dislocations [44]. Since the complex stacking fault energy for ordinary dislocations in $\gamma$ TiAl is quite large and dislocations are very compact, a quasi-equilibrium approach appears to be an appropriate assumption.

4. Rosenthal's analysis for calculation of vacancy super-saturation (or under-saturation) is valid only for jogs moving in an infinite homogeneous medium. A realistic boundary condition might require the vacancy concentration at a finite distance from the moving jog to reach the equilibrium value. However it has been shown that that vacancy concentration is not significantly affected when one changes from infinite to finite realistic boundary conditions [40]. Again, a realistic material is not homogeneous and even the equilibrium vacancy concentration in the material may not be the same everywhere. For instance a jogged-screw dislocation trailing another
jogged-screw dislocation will see a different vacancy concentration. These complications have not been treated here. The jog on the screw dislocation has been considered to be a stand-alone entity and thus unaffected by the presence of other jogs. Qualitatively, it may be argued that the vacancy concentration would rapidly homogenize at these high temperatures.

5. Diffusion is assumed to be primarily via lattice diffusion. Preferential vacancy diffusion along the length of the dislocation core (pipe diffusion) is ignored. This is supported by the activation energy data [Figure 3.8] for steady state creep which suggests lattice diffusion rather than pipe or grain boundary diffusion. The majority of dislocations were observed to be strongly screw oriented. Though pipe diffusion would still be possible, it appears that the extent of pipe diffusion in this case would be significantly lesser than in the case where the microstructure is dominated by edge dislocations. Pipe diffusion along a screw dislocation has been proposed to be more difficult than pipe diffusion through the core of an edge dislocation [49]. In the case of an edge dislocation (whose strain field has a dilatational component) there is a segregation of vacancies to the core. Resultantly, the activation energy for edge dislocation diffusion is smaller than that for screw dislocation, since it no longer contains the energy of formation of the vacancy. The dilatation can be expected to assist easy migration in an edge dislocation [50]. The mobility of vacancies along the screw dislocation has also been investigated [49]. It was observed that a vacancy sitting on a screw dislocation dissociated to the form of extended defects (extended helices). This extension has two negative impacts on the mobility of the vacancy along the screw dislocation. Firstly, the motion of the extended defect involves the
cooperative movement of a large number of atoms, which creates an entropic barrier to easy migration. Secondly, it has been suggested that all atomic motion within individual screw dislocations would be completely correlated and this would not lead to a net flux along the dislocation. All this suggests that a microstructure dominated by screw dislocations will not contribute significantly to pipe diffusion.

6. Short circuit diffusion of vacancies from a positive jog to a negative jog (via lattice diffusion) is not addressed. Two neighboring jogs were measured to be hundreds of nanometers apart. These distances are orders of magnitude larger than typical diffusion lengths at times corresponding to the creep velocity. So it appears valid to assume that the jog picks up and dumps vacancies wholly from the bulk.

3.5. Evaluation of the creep rate under this model

The aim of the previous exercise was to evaluate a relation between jog velocity, jog height and the vacancy super-saturation (or under-saturation) thus created. To this effect we have equation (3.31), which is restated below for a vacancy-producing jog.

\[ C_p - C_0 = \frac{v \cdot \delta(h)}{4 \pi D_v b^2} \] (3.31)

This can be rewritten thus:

\[ \frac{C_p}{C_0} = \frac{v \cdot \delta(h)}{4 \pi D_v b^2 C_0} + 1 \] (3.36)

The above result can be used along with equation (3.9) to give the following result for a vacancy producing jog

\[ \exp\left(\frac{\tau \Omega}{kTh}\right) = \frac{v \cdot \delta(h)}{4 \pi D_v b^2 C_0} + 1 \] (3.37)
By rearranging the terms, we can easily write an equation for jog velocity for a vacancy-producing jog

\[ v = \frac{4\pi D_s b^2 C_0}{\delta(h)} \left[ \exp\left(\frac{\pi\Omega}{kT\theta}\right) - 1 \right] \]  

(3.38a)

A similar equation can be derived for a vacancy-absorbing jog

\[ v = \frac{4\pi D_s b^2 C_0}{\delta(h)} \left[ 1 - \exp\left(-\frac{\pi\Omega}{kT\theta}\right) \right] \]  

(3.38b)

An equal number of vacancy-producing and vacancy-absorbing jogs can be assumed. It will be assumed that, the velocity of the dislocation containing an equal number of positive and negative jogs is the average of the two equations (3.38a) and (3.38b) above. This average leads to the following equation

\[ v = \frac{4\pi D_s b^2 C_0}{\delta(h)} \left[ \sinh\left(\frac{\pi\Omega}{kT\theta}\right) \right] \]  

(3.39)

We then carry out further simplifications based on the following relations [46]:

\[ C_0 = \frac{x_0 f}{a_0^3} \]  

(3.40)

where \( x_0 \) as defined earlier is the atomic fraction of vacancies, \( f \) is the number of atoms per unit cell and \( a_0 \) is the lattice parameter. We already know that the lattice self diffusion coefficient \( D_s \) is related to \( D_v \) via equation:

\[ D_s = x_0 D_v \]  

(3.41)

Combining equations (3.39), (3.40) and (3.41) we can come up with a simplified velocity law as shown:

\[ v = \frac{4\pi D_s b^2 f}{\delta(h) a_0^3} \left[ \sinh\left(\frac{\pi\Omega}{kT\theta}\right) \right] \]  

(3.42)
Since we know that for ordinary dislocations in $\gamma$-TiAl, $b=\sqrt{2}a_0$ and $f=4$, we can further simplify the above equation as shown:

$$v = \frac{4\sqrt{2}\pi D}{b\delta(h)} \left[ \sinh \left( \frac{\tau \Omega}{kTh} \right) \right]$$ (3.43)

The steady state creep rate can be easily related to the steady state velocity via the Orowan equation (Eq (1.4)):

$$\dot{\gamma} = \rho . b . v$$ (1.4)

where $\rho$ is the dislocation density, $b$ is the Burger's vector and $v$ is the dislocation velocity. We can similarly write the Orowan equation for this case:

$$\dot{\gamma} = \rho . \frac{4\sqrt{2}\pi D}{\delta(h)} \left[ \sinh \left( \frac{\tau \Omega}{kTh} \right) \right]$$ (3.44)

We can convert from shear to tensile values of stress and strain-rate using $\dot{\varepsilon} = \dot{\gamma} / 2$ and $\tau = \sigma / 2$. With the knowledge of microstructural parameters, such as dislocation density, jog spacing and jog height, this equation can be used to predict the steady state shear strain rate given an applied shear stress. However, before proceeding any further, we need to examine the functional dependency of the various microstructural parameters on stress. The following sections delve deeper into the determination of these functional relationships. Attempt will also be made to provide quantitative and qualitative evidence for the observed values of these parameters.

### 3.5.1. Dislocation Density

The dislocation density is usually related to stress through the Taylor's equation (Eq (1.5)). Taylor relationship for the stress-dependence of dislocation density has been found
to hold for a variety of materials under creep conditions [51]. It has been found for both class M and class I alloys. The latter case is perhaps most analogous to the present discussion as dislocation mobility is thought to control deformation as opposed to substructure formation.

The dislocation density is quantified by TEM observations. The total length of curved dislocations (curvature was assumed to imply mobility) is calculated from the TEM micrographs. The sample thickness is estimated using CBED techniques. The dislocation density is calculated as the total dislocation length per thickness of the sample. The Schmid factor and the resolved shear stress is calculated with the knowledge of the loading axis relative to the foil normal, and the actual slip system. Figure 3.27 shows the variation of dislocation density as a function of the resolved shear stress. A power-law stress dependence of 2 suggests that Taylor's relationship is a valid assumption for equiaxed γ-TiAl. The value of Taylor factor $\alpha$ was found to be 2, which is identical to the value found by Viswanathan et al [8].

3.5.2. Jog Spacing

The jogged screw model of Barrett and Nix [40] and Viswanathan et al [8] assumed that the jog spacing is invariant with stress. In both models, the physical basis of this assumption is not satisfactorily explained. Additionally, it was noted that the average lamellar spacing used in the latter model (200 nm) is similar in value to the Orowan bowing diameter (for that particular resolved shear stress). This observation provided us the motivation to investigate the possible effect of stress on jog spacing.
The jog moves non-conservatively along with the dislocation. It can also move conservatively, along the length of the screw dislocation in the cross-slip plane. This motion being pure glide would be relatively easy. Several authors have suggested that the screw dislocation moves forward by an "unzipping" process which removes the jogs by purely conservative lateral motion as shown in Figure 3.28 [52-54]. Models for predicting the yield stress anomaly have been developed on this basis.

In the jogged screw model, the jog spacing is assumed to be uniform. It is however logical to assume that a spectrum of spacings is present (a gaussian fit appears appropriate). In such a scenario, unbalanced line tension forces are created that drives the conservative motion of jogs. Due to this motion, jogs spaced close to each other tend to come closer and vice-versa. This in effect leads to jog coalescence (or annihilation, depending on the sign of jogs) and in essence jog coarsening [Figure 3.29].

It is apparent that there are complications involved that can only be treated statistically. To this effect, we have constructed a dynamical simulation of the motion of the dislocation by a simple line tension model. The schematic of the simulation is shown in Figure 3.30. In this simulation, we create an initial dislocation configuration that consists of unit jogs randomly signed and present randomly along the length of the screw dislocation. Typical simulations involve 50 unit jogs initially separated 20 +/-10 nm. A few simulations were initialized with tall jogs (8 Burger's vector) separated by 50+/-20nm.

The rules of the model are given below:

1) At each time step, the position of the jog is updated using the lateral and forward velocities. Simple line tension arguments are used to evaluate these velocities. The
lateral conservative velocity $V(x)$ is related to the net resolved lateral force and the non-conservative velocity $V(y)$ is related to the net resolved climb force [See Figure 3.30]. The relevant equations are given below:

\[
F(x) = T \sin(\theta) - T \sin(\phi) \quad (3.45)
\]

\[
F(y) = T \cos(\theta) - T \cos(\phi) \quad (3.46)
\]

\[
V(x) = \frac{(F(x) - f_p)}{\beta \cdot h} \quad (3.47)
\]

\[
V(y) = \frac{4 \sqrt{2} \cdot \pi \cdot D}{b \cdot \delta(h)} \left( \exp\left( \frac{F(y) \cdot \Omega}{b \cdot h \cdot k \cdot T} \right) - 1 \right) \quad (3.48)
\]

\[
V(y) = \frac{4 \sqrt{2} \cdot \pi \cdot D}{b \cdot \delta(h)} \left( 1 - \exp\left( - \frac{F(y) \cdot \Omega}{b \cdot h \cdot k \cdot T} \right) \right) \quad (3.49)
\]

where $F(x)$ is the net resolved lateral force, $F(y)$ is the resolved forward force, $\phi$ and $\theta$ are angles subtended by the dislocation at the cusp, $V(x)$ is the lateral conservative velocity, $f_p$ is the frictional resistance to conservative motion [13], $\beta$ is the glide mobility, $h$ is the jog height, $V(y)$ is the non-conservative velocity. The other terms have been defined earlier. The line tension $T$ is assumed to be $G.b^2/2$ [44]. The angles $\phi$ and $\theta$ are calculated from the positions of the adjoining jogs and the bowing radius $r$. The bowing radius is related to the applied stress: $r = G.b/2 \tau$ [44]

2) Periodic jog-pair nucleation events are allowed (through double cross-slip). The distance between the jog-pair generated is random. The jog-pairs grow, only if they are separated by a fixed value (picked to be 10nm)

3) When two jogs of the same sign come together, they form a super jog; oppositely signed jogs annihilate each other.
4) The dislocations are bowed to perfectly circular segments between the jogs - the radius depending the applied stress. Widely separated jogs have long straight near-screw segments between them.

5) No dislocation interaction is considered.

The typical evolution of the jogged-screw dislocation is shown in Figure 3.31. The dynamic simulations predict a coarsening of jog spacing due to lateral motion of the jogs, as expected. It also predicts the evolution of jog spacing and jog height [Figure 3.32 and 3.33]. It is seen that the average jog spacing coarsens and reaches a "steady-state" value very rapidly [Figure 3.32]. The jog spacing does not keep increasing because the very coarse jog spacings have a higher probability for jog nucleation and this event refines the jog spacing.

Since jog spacing evolution is so rapid, this process may not be significant in determining the primary creep transients. In the same token, the simulation results also lead us to believe that the rate of jog nucleation (and the evolution of jog height) may be critical for predicting the transient behavior. The most significant result that comes out of these simulation studies is that the jog spacing is inversely proportional to the applied stress [Figure 3.34].

To confirm this prediction, jog spacing measurements were made in the TEM, for a range of stresses. The jogged-screw dislocations were imaged looking normally at the slip plane (beam direction parallel to the slip plane normal). So no corrections had to be made to the spacings measured. Figure 3.35 shows the jog spacing distribution as a function of stress. It is evident that the distribution shifts to smaller values as the stress is increased. The average jog spacing is also plotted as function of stress (in Figure 3.36). It
is very clear that the jog spacing is inversely proportional to the applied stress, as predicted by the simulations. The jog spacing dependence indicated in equation (3.50) on stress derived from actual TEM observations is used in equation (3.44)

\[ l = K\tau^{-1} \]  

(3.50)

In the next section we will explore the possible stress dependence of jog height.

### 3.5.3. Critical Jog height

A distribution of jog heights has been observed by Sriram et al and Viswanathan et al [8, 13]. The model by Viswanathan et al., assumed that there exists an upper bound to the height of a "draggable" jog. Jog coalescence events, as described in the last section could give rise to an increase in jog heights [8]. It is assumed that when the jog became taller than the critical height, it stops being dragged and starts acting as a dislocation source as shown in Figure 3.37 (as the near-edge segments attached to the jog bypass each other). This critical height is based on the stress required to break an infinitely long pure edge dipole. The critical jog height is given by:

\[ \tau_{\text{bypass}} = \frac{G.b}{8\pi(1-\nu)h} \]  

(3.51)

TEM observations by Viswanathan et al [8], however suggests that jogs much taller than this limit, appear to be in" dragging" configuration and not in a "double-ended" source configuration. The authors explain these observations on the basis of elastic anisotropy, Peierl's stress effects and the cusp configuration. Though these could be probable causes, substantial proof has not been provided. In view of these discrepancies, the issue of critical jog height is reviewed and modified.
There are three distinct scenarios possible as a function of jog height (Figure 3.37). When the jog is not too tall, it is relatively easy to drag the jog. As the jog height increases, the stress required for dragging the jog increases significantly and it becomes easier to lay a dipole instead. When the jog grows even taller, the applied stress is actually sufficient to break the dipole and cause the dislocation segments to bypass and the jog could then act as a source. Figure 3.38 indicates TEM micrographs with jogs in all three configurations. Also seen are prismatic loops that may have formed behind dipoles. A complete thermodynamic assessment of the critical height involves a comparison of the stress required for each of these processes. The following equations are used to evaluate the minimum stress (as a function of height) that needs to be overcome to drag a jog, extend a dipole or cause dipole bypass.

\[
\tau_{\text{dipole}} = G_b \left( \frac{1}{2\pi(1-\nu)l} \ln \left( \frac{h}{b} \right) + \frac{E_{\text{core}}}{lb^2} \right) \quad (3.52)
\]

\[
\tau_{\text{bypass}} = G_b \left( \frac{1}{8\pi(1-\nu)h} \right) \quad (3.51)
\]

\[
\tau_{\text{jog}} = \frac{h.k.T[\arcsin h(\frac{v.b.\delta(h)}{4\pi.D.f})]}{\Omega.l} \quad (3.53)
\]

While the former two processes are athermal, jog dragging is not only dependent on the temperature, but also on the jog velocity. Dragging the jog becomes increasingly favorable with increasing temperature, and at smaller velocities. Figure 3.39 shows the minimum stress required for each of these processes at different jog velocities and temperatures. By comparison of the minimum stresses required to initiate each of these processes, it is clear that at creep velocities and higher temperatures, the jog directly goes from being dragged to a dipole bypass mechanism, as the jog height increases.
The jog velocity at each stress level can be calculated from the stress-steady state strain rate data using Orowan's equation (Equation (1.15)). The cross over from jog dragging to dipole bypass for this jog velocity can be found by iteratively solving equations 3.51 and 3.53 for the value of $h$. In a similar fashion, the jog velocity can be extracted for each stress level and the critical jog height can be found [Figure 3.40 and Figure 3.41].

$$h_d = L\tau^a$$ \hspace{1cm} \text{where } a = -2 \text{ to } -3 \hspace{1cm} (3.54)

Interestingly, the critical jog height still varies inversely with stress, as we assumed by Viswanathan et al [8]. However, the "modified" critical height has a much higher stress dependence that predicted earlier (proportional to $\tau^{-2.0-3.0}$). This difference is due to the following reasons:

a) The earlier approach does not consider jog dragging and dipole bypass to be competing processes. So the critical jog height found by Viswanathan et al has no temperature (or strain-rate) dependence [8]. However, at creep temperatures and strain rates, diffusion mediated processed such as jog dragging become relatively easier than an athermal process like dipole bypass.

b) The dragging of jogs is further made easy because of the FLML assumption due to which taller jogs drag more easily than previously anticipated by the CMP assumption.

Further support for this approach comes from the fact that the critical jog heights predicted are much taller than predicted earlier and as seen in Figure 3.40, that the measured jog heights (for one stress level) compares very well with the predicted.
The stress dependencies of dislocation density, jog spacing and jog height can now be incorporated into 3.44

3.6. The Modified Jogged-Screw Model

Equations (1.5), (3.50), and (3.54) are restated below.

\[
\rho = \left( \frac{\sigma}{\alpha \cdot b \cdot G} \right)^2, \text{ where } \alpha = 2
\]

(1.5)

\[
l = K \tau^{-1}
\]

(3.50)

\[
h_d = L \tau^n
\]

(3.54)

These equations can be used along with equation (3.44), to predict creep rates.

Since height of jogs range from a Burger's vector to the critical jog height, it is important to know which value is best representative of the distribution. This characteristic jog height is picked to be a constant fraction of the critical jog height though a parameter \( \beta \), as shown below

\[
h = \beta h_d
\]

(3.55)

Qualitative arguments are provided for a suitable choice for \( \beta \)

A) If a rate process (like creep) consists of a series of parallel, independent steps, then the rate is controlled by the fastest step. However if it consists of many steps that are in series, then it is the slowest process that is rate limiting. In the case of a single jogged screw dislocation, the velocity of each jog will be independent of each other, at the early stages of creep. However, as the faster jogs start pulling away from the slower jogs, it is at the cost of increasing dislocation line length. Line tension would work to partition the forces on the two jogs such that the faster one slows down and the slower one catches up
and the line length is not increased any further. At steady state, it can be envisioned that the dislocation would move self similarly at one velocity. TEM observations of crept samples seems to indicate that jogs on one screw dislocation indeed move cooperatively. It is evident that at steady state, the dragging of each jog is coupled with its neighbors. It thus appears that the strain rate would be controlled by the slowest step.

B) Creep deformation in equiaxed γ-TiAl is proposed to take place through a series of steps:

- Loops of ordinary dislocations are generated from sources in the interior of the grain or at the grain boundaries.
- The edge segments with greater mobility run out, leaving behind the sluggish screw segments.
- The screw segments undergo frequent double cross-slip and develop jogs.
- The leading screw dislocation is the slowest, since the average jog height on this dislocation is the largest.
- In a "pile-up" type mechanism, the operation of the dislocation source is rate limited by the velocity of the leading screw dislocation.
- The lead dislocation develops jogs that are tall enough to act as sources. The process is repeated all over.
- At steady state, the equiaxed grain is saturated with dislocations, the velocity of which and the operation of all sources, is limited by the velocity of the tallest jog.

Dislocation microstructure [Figure 3.9] suggests that such a mechanism is indeed probable. The rate-limiting step is clearly the rate of dragging of the slowest/tallest jog.
From both arguments, the choice of $\beta=1$ seems most appropriate. The value of $\beta=1$ is a statement that the tallest jogs (and hence the slowest) are the rate-controlling jogs along a given dislocation. Combination of equations (1.5), (3.50), (3.54) and (3.44) leads to creep rate predictions that are in excellent agreement with the observed creep rates for two different temperatures [Figure 3.43 and Figure 3.44]. In this model, there are now no unsubstantiated assumptions, and no fitting parameters. There in lies the simplicity and the rigidity of the model. The application of this model to $\alpha$-Ti has been shown in Appendix 2.

3.7. Conclusions and Unresolved issues

Based on the observation of cusped $1/2[110]$ ordinary screw dislocations, the jogged-screw model was adopted, where the rate-controlling step is assumed to be the non-conservative dragging of the jogs. The original solution has been reformulated to take into account the finite length of the moving jog. By accounting for the fact that there are three competing mechanisms (jog dragging, dipole dragging, dipole bypass) possible, we can arrive at a modified critical jog height beyond which the jog is not dragged. Dynamic simulation using a line tension model and TEM measurements confirm that the jog spacing is inversely proportional to the applied stress. Taylor’s expression for the dislocation density is confirmed by actual dislocation density measurements. Combining all of these parameters and dependencies, derived both from experiment and theory, leads to an excellent prediction of creep rates and stress exponents.

The effect of temperature and stress on the evolution of jog heights is still intriguing. An increase in temperature should lead to an increase in the pinning point
density, the implications of which are not quite clear. This may lead to a larger activation energy owing to additional temperature dependent parameters in the jogged screw model. Since the jog spacing does not depend on temperature, this increase in the number of cross slip events appears to affect only the primary transient behavior. Jog height dependence on stress needs to be characterized using stereo-pair imaging TEM techniques. The shape of the creep transients is unlike that of a "typical" glide controlled creep mechanism. A detailed study of the evolution of model parameters with strain will provide important clues towards understanding the transient behavior. Such an understanding will aid in the dynamic simulation efforts.
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Figure 3.1. The creep curves for the equiaxed microstructure at different stress levels. The creep transient is “normal”.

Figure 3.2. The same curves as in Figure 3.1, replotted on a log-log scale. The power-law time exponent is between 0.3 and 0.5.
Figure 3.3. Several creep tests conducted under the same creep conditions. The creep behavior for these tests is virtually identical.

Figure 3.4. The minimum creep rate is plotted as a function of stress for two different temperatures. The stress exponent is between 5 and 6.
Figure 3.5. Creep tests conducted at different temperatures at 300 MPa

Figure 3.6. The minimum creep rate plotted as a function of 1/T for different strain levels
Young’s Modulus as a function of temperature

\[
\frac{dE}{dT} = -4.648 \times 10^{-7} \text{ Pa/K}
\]

Figure 3.7. The drop in Young’s Modulus as a function of temperature [9]

**Ti-48 Al (Eq), Compression crept at 300 MPa**

- Activation Energy for Lattice-Diffusion
- Activation Energy for Pipe- or Grain Boundary-Diffusion

Figure 3.8. The value of the “true” creep activation energy plotted as a function of strain and compared to activation energy for lattice self diffusion \(Q_d\) (295kJ/mol) and the activation energy for pipe/grain boundary diffusion (from ref [12])
Figure 3.9 TEM micrograph showing microstructure after creep at 1041 K and 207 MPa. The presence of cusped screw dislocations is evident [8].

Figure 3.10 Dark field TEM micrograph (g = 202) showing microstructure after creep at 1033K and 300 MPa. Numerous cusped screw dislocations and loops are seen.
Figure 3.11 TEM micrograph showing microstructure after creep at 1033K and 300 MPa. Jogged-screw dislocations and dislocation debris observed \( (g = 202) \).

Figure 3.12 Cusped jogged-screw dislocation structures observed by a) Ott [15], and b) Morris et al. [14]
Figure 3.13 Cusped jogged-screw dislocation structures observed by a) Appel et al [16], and b) Sriram et al. [13]
Figure 3.14. Schematic showing the formation of a jog by a double cross-slip process.

Figure 3.15. Jogs are not formed when the kink expansion rate is much larger than the kink nucleation rate.
Figure 3.16. The interaction of two kinks lying on different \{111\} planes can give rise to the formation of unit jogs. This requires that the kink nucleation rate be comparable to the kink expansion rate.

Figure 3.17. Jog-jog interaction will not give rise to the formation of tall jogs if there is an equal number of positive and negative unit jogs.
Figure 3.18. A possible scenario for the formation of tall jogs. The dislocation can cross-slip in both the forward and backward directions in an attempt to avoid the obstacle (adapted from ref [32]).

Figure 3.19. The advance of a self-similarly moving jogged screw dislocation by a distance $a$. The force balance at steady state is indicated.
Figure 3.20. The vacancy concentration behind the jog is the superposition of vacancy concentration due to a linear string of \( n \) point sources.

Figure 3.21. The concentration profiles around the jog under the CMP and FLML assumptions. The scale of the x and y axis is in \( n \), number of Burger’s vectors.
Figure 3.22. The actual value of super-saturation at point O in Figure 3.20, as a function of jog height under the CMP and FLML assumptions.

Figure 3.23. Orders of magnitude change in the jog velocity affects the correcting function only marginally, indicating the insensitivity of this function to jog velocity.
Figure 3.24. Insensitivity of the correcting function to lattice diffusivity

Figure 3.25. Insensitivity of the correcting function to \( v/D_v \) ratio
Figure 3.26. The supersaturation limit is seen to change very little with orders of magnitude change in the $v/D_v$ ratio.

Figure 3.27. The variation of dislocation density as a function of the resolved shear stress confirms Taylor’s relation.
Figure 3.28. “Unzipping” process which removes the jogs by purely conservative lateral motion [5]

Figure 3.29. A schematic of the jog spacing coarsening process due to the conservative motion of jogs driven by line tension. The X’s denote positive jogs and O’s denote negative jogs. Jog coalescence, annihilation and jog-pair nucleation events are indicated.
Figure 3.30. A schematic of the dynamic simulation of jogged-dislocation motion
3.31. The result from a part of one simulation is shown. Evolution of the jogged-screw dislocation and jog spacing coarsening are observed. An overall reduction in the total number of jogs (accompanied by an increase in jog height) is also noted. This simulation was run at 200 MPa, 815 C, with an initial configuration consisting of 50 randomly-signed unit jogs initially separated 20 +/-10 nm. The scale is in meters.
Figure 3.32. Jog spacing evolution with time is indicated for different stress levels. The “steady-state” jog spacing is inversely proportional to the applied stress. Jog spacing coarsens rapidly.

Figure 3.33. The evolution of jog height with time is indicated.
Figure 3.34. The simulations predict that the jog spacing is inversely proportional to the applied stress.

Figure 3.35. Jog spacing distribution at different stress levels derived from actual TEM measurements.
The average jog spacing is measured to vary inversely with stress as predicted by the simulations.

Figure 3.37 There are three distinct scenarios possible as a function of jog height. Jog dragging at short jog heights, dipole dragging at intermediate jog heights and dipole bypass at large jog heights.
Figure 3.38. TEM micrographs with jogs in jog dragging, dipole dragging and dipole bypass configuration (single-ended source). Also seen is the formation of prismatic loops, that may form behind dipoles. Sample crept to 1% at 300 MPa and at 760 °C.

Figure 3.39a. The minimum stress required for each of the processes at different jog velocities.
Figure 3.39a. The minimum stress required for each of the processes at different temperatures.

Figure 3.40. The modified critical jog height is compared to the one used by Viswanathan et al. [8], as a function of stress at 1041 K. Actual measurements of jog heights indicated by the hollow circles.
Critical Jog Height (from Equation (3.51))

Modified Critical Jog Height (Temp =\(1088\) K)

**Critical Jog Height**

**Stress (MPa)**

Slope = -2

![Graph](image)

Figure 3.41. The modified critical jog height is compared to the one used by Viswanathan et al. [8], as a function of stress at 1088 K.

**Model Predictions**

*Model predictions* (\(\beta=1, \alpha=2\))

![Graph](image)

Figure 3.42. Model predictions at 1088K compared with experimental values
Figure 3.43. Model predictions at 1041 K compared with experimental values
4. CREEP IN FULLY LAMELLAR $\gamma$-TiAl

4.1. Phenomenology of creep in lamellar microstructures.

Typical creep curves for the binary Ti-48Al (FL) alloy are indicated in Figure 4.1. These were crept at 207 MPa and 815°C under tension. The creep curve for two different lamellar spaced alloys is indicated. The sample with the finer lamellar spacing creeps considerably slower. (Note: It will be discussed later that ascribing an average lamellar spacing value may not be a sufficient representative of the lamellar distribution). Creep tests were also conducted on the K5 alloy under compression at different stresses and temperatures. A typical creep curve of the K5 alloy is shown in Figure 4.2. Figure 4.3 indicates two different tests conducted under the same conditions. Consistency of creep results between different samples is acceptable.

Figure 4.4 shows the creep curve for the binary alloy at two different temperatures. Assuming Dorn equation and using the minimum creep rate at the two temperatures (same stress level) the creep activation energy was calculated and found to be 272 kJ/mol. The same procedure was employed for the K5 alloy. The activation energy was calculated at one high stress level (315 MPa) for the temperatures of 760°C and 815°C. This was found to be to be 308 kJ/mol. The activation energy was also calculated at a low stress level (150 MPa) through a series of temperature change tests and was found to be 360 kJ/mol [Figure 4.5]. Accounting for the change in Young's
modulus (with temperature), we can shave off approximately 25kJ/mol from the creep activation energy values to get the true activation energy. At moderate to high stresses, the activation energy calculated is very close to the Ti self-diffusion coefficient (= 295kJ/mol). The low stress value is slightly higher than this. The possible effect of Nb, W and Cr, present in the K5 alloy cannot be ruled out. Since the creep activation energy is not too different from the lattice self-diffusion coefficient, it is assumed that creep in both these alloys is mediated by lattice diffusion at all stresses.

The creep curves for both the binary and K5 alloy is seen to exhaust initially during primary creep. Both alloys exhibit a very short minimum creep-rate regime, before accelerating. It is to be noted that the K5 samples were tested in compression and yet exhibited tertiary creep behavior (the possible origin of this behavior is discussed in the next chapter). The strain rate versus stress curves for the lamellar Ti-48Al is indicated in Figure 4.6. These values are obtained through a series of stress jump tests. Also shown for comparison is the creep behavior of the equiaxed binary alloy and the K5 FL alloy. There are several features of the creep response of FL alloys that is significantly different from that of the equiaxed alloy:

- The creep rates for the lamellar alloys is much lower than that for the equiaxed alloy
- There appears to be two different regimes: a low stress regime with a stress exponent between 2 and 3 and a high stress exponent where the stress exponent is between 7 and 8. This two stress-regime behavior of the FL alloy has been frequently reported as shown in Figure 1.14 [1-3].
- There seems to be an effect of lamellar spacing: reducing the lamellar spacing is seen to reduce the creep rates (and increase the stress exponent in the high stress regime).
The K5 alloy appears to have a much better creep response when compared to the binary alloy. It is important to recognize that the lamellar spacing is significantly finer for the K5 series alloys than for the binary case.

From Figure 4.6, it is obvious that FL alloys are of greater engineering interest than the equiaxed alloys (at least from a creep resistance perspective). Hence there is a need for lucid understanding of the underlying creep mechanisms and for a physically based predictive model. Any attempt at developing a creep model for FL microstructures has to be able to predict the three features seen in the strain rate-stress curve. Besides being phenomenological, the model also has to take into account the observed crept microstructures. So, as with the equiaxed microstructures, we begin by looking at the deformation microstructures. We will first concentrate on samples deformed in the “high-stress” regime.

4.2. Deformation microstructure

Samples crept to minimum creep rate, at stresses in the high-stress regime supply ample microstructural evidence of $1/2[110]$ ordinary dislocation activity within the $\gamma$-laths of the lamellar structures. There is very little evidence for twinning or subgrain formation at strains corresponding to minimum creep rate. The extent of twinning (trans-lamellar and parallel) and subgrain formation appears to increase with increasing strain. Even at very large strains, the subgrain formation is restricted to the widest laths [Figure 4.7]. Lamellar interfaces of both types ($\gamma'/\gamma$ and $\gamma/\alpha_2$) appear to effectively constrain deformation to individual $\gamma$-laths, with little evidence for direct transmission under creep conditions. In light of the fact that $\alpha_2$ is considered to be a stronger phase at these
temperatures and it accounts for only 7% of the total volume, it is proposed that dislocation activity within the γ-laths may be very significant in controlling the primary and steady state creep response. This assumption has been previously made by several authors and is also supported by evidence that the creep rates are not affected by the $\alpha_2$ volume fraction [4-6].

The other characteristic of deformation in lamellar microstructures is that it is highly inhomogeneous, with smaller dislocation densities in the thinner laths and significantly larger densities in the thicker laths (Figure 4.8). The thinnest lamellae are free of dislocations, lamellae of intermediate width have hard-mode dislocations channeling through them, while the thicker lamellae have near-screw dislocations that are often cusped in configurations similar to those in equiaxed structures. Such inhomogeneity in deformation microstructure, and its possible role in increasing the stress exponents in lamellar structures, has been suggested earlier [3].

Figures 4.9, 4.10 and 4.11 show some of these cusped near-screw dislocations in the binary alloys and in the K5 alloy. While most dislocations appear to be hard-mode type dislocations, some trans-lamellar soft mode dislocations are also observed in the cusped configuration [Figure 4.10]. Almost all hard-mode dislocations are exclusively ordinary dislocations, as is clear by g,b analysis (Figure 4.12). Though a detailed stereopair technique has not been used in this case, the series of pictures in Figure 4.13 clearly indicates that the cusps are indeed associated with jogs. Tilting the sample parallel to the lamellar interface has shown that hard-mode screw dislocation lays out near-edge dislocation segments along the interface [Figure 4.14].
The observation of cusps on the ordinary dislocations suggests that the jogged-screw model mechanism could indeed be operational in FL alloys too. In the next section we will try to develop the jogged-screw model for lamellar microstructures.

4.3. Jogged-Screw model for FL microstructures

It was shown in Figure 4.6 that there is a significant effect of lamellar spacing on the creep behavior of FL alloys. In Orowan's equations, either the dislocation velocity \( v \) or the dislocation density \( \rho \), or both, have to be a function of lamellar spacing to account for this effect. These effects are explored in the subsequent sections. (Note: We develop this model based on hard-mode screw dislocation, the explanation for this assumption is discussed in detail in a later section)

4.3.1. Dislocation velocity and its dependence on lamellar spacing

In this section we will proceed with the development of a velocity law in a fashion identical to that employed for equiaxed structures in Section 3.5.

As a jogged dislocation moves forward by the non-conservative dragging of jogs, it lays out near-edge segments along the bounding interfaces (both \( \gamma/\gamma \) and \( \gamma/\alpha_2 \)). So, at steady state, the work done by the applied shear stress in moving the dislocation forward by \( a \), namely \( W_{\text{Shear}} \), is balanced by the work done by the attractive forces due to the creation of vacancies (due to the non-conservative motion of jogs), \( W_{\text{Chem}} \) and the work done in extending the dislocation segments laid out along the interface, \( W_{\text{Dipole}} \) (see Figures 4.15 and 4.16).
For a vacancy producing jog, the following expressions have been used for these work terms (from Eqs (3.5) and (3.7) for equiaxed microstructures):

\[
W(\lambda)_{chem} = n(\lambda') \cdot f_p \cdot a = n(\lambda') \cdot \frac{kTbh}{\Omega} \ln \frac{C_p}{C_0} \cdot a \tag{4.1}
\]

\[
W(\lambda)_{dipole} = a \cdot E_{dipole} \tag{4.2}
\]

\[
W(\lambda)_{shear} = \tau \cdot b \cdot \lambda' \cdot a \tag{4.3}
\]

The meaning of the terms is exactly the same as for equiaxed microstructures. The new terms are defined:

- \(a\) is the distance that the dislocation moves forward by when it produces one vacancy. This can be easily computed and is equal to \(\Omega/bh\), where \(\Omega\) is the atomic volume and \(h\) is the jog height

- \(n(\lambda')\) is the number of jogs present in a screw dislocation length of \(\lambda'\). Due to the geometry of the lamellar structures, the hard mode glide plane is oriented at 71° to the interface place. Also the [110] direction is at 60° to the interface. Using this knowledge \(\lambda'\) can be calculated:

\[
\lambda' = \frac{\lambda}{\sin(71\degree) \cdot \sin(60\degree)} \tag{4.4}
\]

\(\lambda'\) is approximately \((1.22\lambda)\).

- \(E_{dipole}\) is the energy required to extend of a dipole that is separated by a distance \(\lambda\) and is numerically given by:

\[
E_{dipole} = \frac{G \cdot b^2 \cdot \Psi(\theta)}{2 \cdot \pi(1 - v)} \cdot \ln \left(\frac{\lambda'}{b}\right) + \frac{2E_{core}}{b} \tag{4.5}
\]
• \( \Psi(\theta) \) is a correction factor for accounting for the fact that the segments are near-edge (and not pure edge). \( \theta \) is the angle made by the Burger's vector with the interface. \( \Psi(\theta) \) is given as:

\[
\Psi(\theta) = \sin^2(\theta) + \frac{1}{(1-v)} \cos^2(\theta)
\]  

(4.6)

In this case, \( \theta = 60^\circ \) and \( \Psi(\theta) = 1.107 \) (for a Poisson's ratio of 0.3)

• \( E_{core} \) is the core energy of the dislocation. This value is assumed to be 2.2 eV [7].

At steady state we know that \( W_{Shear} = W_{Chem} + W_{dipole} \). Using equations (4.1), (4.2) and (4.3), we can determine \( C_p/C_0 \):

\[
\frac{C_p}{C_0} = \exp\left[-\frac{\Omega}{n(\lambda')kTb} (\tau \lambda' - E_{dipole})\right]
\]  

(4.7)

Rearranging the various terms and approximating, the value of \( n(\lambda') = \lambda' / l \), (where \( l \) is the jog spacing), we can write \( C_p/C_0 \) as:

\[
\frac{C_p}{C_0} = \exp\left[\frac{\Omega \lambda'}{kTh} (\tau - \frac{E_{dipole}}{b \lambda'})\right]
\]  

(4.8)

This value can be related to the jog velocity using equation (3.31) as shown below:

\[
\frac{C_p}{C_0} = \frac{\nu(\lambda)\delta(h)}{4\pi D_b \nu^2 C_0} + 1
\]  

(3.31)

The velocity of the vacancy-producing jog can be found by equating (4.8) and (3.31):

\[
\nu(\lambda) = \frac{4\pi D_b \nu^2 C_0}{\delta(h)} \cdot \exp\left[\frac{\Omega l}{hkT} (\tau - \frac{E_{dipole}}{b \lambda'})\right] - 1
\]  

(4.9)

The velocity of a vacancy-absorbing jog can be similarly calculated as:
Making the same simplifications as in the equiaxed case and assuming the velocity of the dislocation to be the average velocity of vacancy producing and vacancy-absorbing jogs, we can write the velocity of the jogged screw dislocation in the lamellar case.

\[
v(\lambda) = \frac{4\sqrt{2} \pi D_s}{b \delta(h)} \cdot \left\{ \sinh \left[ \frac{\Omega l}{hkT} \cdot \left( \tau - \frac{E_{\text{dipole}}}{b \cdot \lambda} \right) \right]\right\}
\]  

(4.11)

It is interesting to note that the equation looks very similar to that for equiaxed microstructures shown below (Equation (3.42)):

\[
v = \frac{4\sqrt{2} \pi D_s}{b \delta(h)} \left[ \sinh \left( \frac{\Omega l \tau}{hkT} \right) \right]
\]  

(4.12)

However, in the lamellar case, the applied stress is now opposed by a "backstress" \( \tau_b \), such that the effective stress is reduced. The backward stress is related to the stress required for channeling \( = \frac{E_{\text{dipole}}}{\lambda' b} \). The origin of backstress during flow in thin channels has been confirmed by several researchers [8-10].

The dependence of the "backstress" \( \tau_b \), and the effective stress \( \tau_{\text{eff}} = \tau - \tau_b \) as a function of lamellar spacing is shown in Figure 4.17. It is evident that decreasing the lamellar spacing decreases the effective stress such that below a critical cutoff, the lamella experiences no driving force.

### 4.3.2. Dislocation density and its dependence on lamellar spacing

Since Taylor's relation for dislocation density is quite universally applicable and has already been shown to be appropriate in equiaxed \( \gamma \)-TiAl, its validity for the lamellar
microstructure can be assumed. However as observed in the deformation microstructure, the dislocation density is heterogeneous, with high densities in the wider laths and smaller densities in the thinner ones. The physical reason for this observation is that dislocation source operation is much more difficult in the thinner laths than in the thicker ones (due to the channeling stress). In light of this it is proposed that the dislocation density is related to the effective stress (and not the applied stress) via a Taylor equation as shown

\[ \rho(\lambda) = \left( \frac{\tau(\lambda)_{\text{eff}}}{Gb\alpha} \right)^2 \]  

(4.13)

The total dislocation length is measured as a function of the lamellar width using TEM techniques. The sample thickness is measured by tilting the sample normal to the lamellar interface. The projected lamellar interface width and its dependence on the tilt angle are utilized to extrapolate the sample thickness. The dislocation density is calculated as the total dislocation length per thickness of the sample. The measurements made on the binary (fine lamellar) alloy are shown in Figure 4.18. It is observed that the dislocation density could indeed be related to the effective stress (rather than the applied stress). The value of \( \alpha \), the Taylor factor was measured to be 1.2.

At this juncture it is possible to calculate the strain rate as a function of stress, for a given average lamellar spacing based on the Orowan equation. However it has been observed that there is always a distribution of lamellar spacing in any FL lamellar alloys. While many researchers report an average lamellar spacing value, Parthasarathy et al have suggested that it may be better to report a "log-normal" average [11]. It is also important to evaluate the effect that lamellar distribution on the creep rates. In section 4.5
we will develop the basis for calculating strain rates when there is a distribution of lamellar spacings. Before getting into that, however, we will attempt to justify some of the assumptions made in developing the model for FL structures, particularly with respect to the choice of hard-mode dislocations for evaluating the velocity law. To this effect, the results from backflow experiments will be presented and analyzed.

4.4. Backflow Experiments

4.4.1 Internal stresses in a FL structure

Microstructural examinations suggest that deformation in lamellar TiAl is inhomogeneous. It also appears that within a colony there are significant back stresses that prevent flow and that these back stresses are a function of the lamellar spacing. In an attempt to strengthen the concept of effective stress, we performed several stress drop tests (also called strain transient dip test [12]).

Typical stress drop tests involve dropping the stress level and monitoring strain evolution with time. Depending on whether final stress level is smaller or larger that the back stress, the material will either flow backwards (driven by the back-stress) or forward (under the forward stress). By conducting a series of these tests with different drops in stress level, it is possible to estimate the back-stress (i.e., when the material does not flow either forward or backward).

Our backflow experiments were conducted on the FL K5 alloy, the Ti-48Al (Eq) alloy and the Ti-48Al (PST, in the two hard and one soft orientation). The aim was to obtain qualitative information about the internal stresses as a function of microstructure and strain rather than get a value for the back stress. The choice of these three sample
types is to compare the behavior of the polycrystalline FL lamellar material with a) equiaxed material (polycrystalline, but no laths) and, b) a PST crystal (lamellar, but no grain boundaries).

In our experiments samples are crept at 300 MPa and 815°C and the stress is dropped to 30 MPa (90% drop) when the creep strain reaches a predetermined value. The backflow behavior is monitored following the stress drop and when the sample stops flowing back (which could be over many hours), it is reloaded to the original stress level and allowed to creep to a second strain level and so on. Five parameters were quantified in each case, for each strain level [see Figure 4.19]:

- The instantaneous strain during a drop. This consists of an elastic contribution and a fast plastic portion (could be due to the bowing back of dislocations to confirm to the lower stress level)
- The instantaneous strain during the jump up. This again consists of an elastic portion and a fast plastic portion. (Note: The elastic strain for a stress drop/jump of 270 MPa is about 1.8.10⁻³).
- The ratio $M'$ between the instantaneous strain during the drop to that during the jump up.
- The ratio $R'$ between the backward rate and the forward rate at time corresponding to the stress drop. The negative sign corresponding to the negative strain rate is dropped.
- The total anelastic (backflow) strain recovered.

These quantities have been compared for the five samples [K5 Alloy (FL), Ti-48 Al (Eq), Ti-48Al (PST, A oriented), Ti-48Al (PST, B oriented) and Ti-48Al (PST, N
oriented) in Figures 4.20 through 4.28. The important results from these tests are discussed below:

- It is evident that the instantaneous drop and jump strains are much more than the elastic strain, indicating that considerable fast plastic deformation takes place [Figures 4.20 and 4.21]. It is to be noted that the FL alloys exhibit a considerably greater fast plastic strain than the equiaxed or PST alloys.

- The values of instantaneous strain, drops with increasing creep strain for all samples [Figures 4.20 and 4.21]. This effect probably arises from the fact that these samples were tested in compression. With increasing strain, the cross-section area increases, and thus the total jump (or drop) $\Delta \sigma$ in the stress value decreases. The change in $\Delta \sigma$ with strain will not have a significant effect on the elastic strain, though the plastic strain change could be substantial (assuming plastic strain during loading to be related to the stress change via a power law). Another source of this effect could be strain hardening which causes a drop in the fast plastic strain with increasing strain.

- The ratio $M'$ is noted to be less than unity for most cases, indicating that there is a larger instantaneous strain during loading [Figure 4.22]. It implies that the fast plastic strain during loading is greater than the fast plastic strain recovered during unloading.

- The ratio $R'$ is a very useful parameter. This ratio between the backward strain rate and forward strain rate at the time of the stress drop is envisioned to be a gauge of the internal or back stress. If the initial and final stress levels are $\sigma_1$ and $\sigma_2$, and the backstress is $\sigma_b$, the forward flow is driven by an effective stress of $\sigma_1 - \sigma_b$, and the backward flow by $\sigma_b - \sigma_2$. The forward flow strain rate is proportional to $(\sigma_1 - \sigma_b)^n$.
and backward flow strain rate is proportional to \((\sigma_b - \sigma_2)^n\). If the forward and backflow mechanisms are the same, \(n \sim n'\), and all other constants cancel out, which leads to the equation for \(R'\)

\[
R' = \left(\frac{\sigma_b - \sigma_2}{\sigma_1 - \sigma_b}\right)^n
\]

(4.14)

It is evident that larger values of \(R'\) implies a large value of the back stress. This parameter is rather useful, since it normalizes the effect of microstructure and for this reason, the value of \(R'\) is assumed to be a direct measure of the internal stress. In Figure 4.23, the ratio \(R'\) for the three PST orientations are compared. It is evident that the N orientation has the maximum \(R'\) value and consequently, the maximum backstress. The back-stress for the other hard orientation (A) is smaller than that for the N orientation, but larger than for the soft orientation (B). The back-stress value for the equiaxed microstructure is compared to that of the FL in Figure 4.23. It is evident that the FL material has a much greater back stress than the equiaxed material. It is also clear that the internal stresses in the FL material (and at a slower rate in the equiaxed material) appear to be building up with strain. Such trends cannot be conclusively established for the PST crystals. If we compare the FL results with that of the PST [Figure 4.25], it appears that early on in creep, the internal stresses in the FL material might correspond to either the B or the A orientation, while at larger strains the FL material behaves more like the N oriented PST crystal.

- Finally, we compare the anelastic strain recovered for the five specimens. Anelastic strain can also be viewed as a measure of the total back stress, greater the back stress, greater is the plastic flow backward. The trends that are observed in Figures 4.26
through 4.28 are very similar to that observed for R'. It is again evident that among the PST crystals, the N orientation has the maximum anelastic strain, and the B orientation has the minimum anelastic strain, with the A orientation falling in the middle [Figure 4.26]. The comparison of FL microstructure and the equiaxed shows that the FL microstructure has much larger anelastic strain than the equiaxed microstructure [Figure 4.27]. The anelastic strains build up with increasing creep strains both cases (in the FL material, the anelastic strain appears to drop slightly in the tertiary creep regime). Comparison of the FL material with the PST material indicates that the FL material behaves similar to the B orientation PST crystal at low strains but with increasing strain behaves more like the hard N oriented PST crystal [Figure 4.28].

In the next section, we will justify the assumption that the minimum creep rate in FL lamellar material is controlled by the hard oriented grains, on the basis of the above observations and support from literature

4.4.2. Effect of colony size, lamellar spacing and orientation

Schmid factor considerations [Table 4.1] suggest that a soft oriented PST should be dominated by soft-mode dislocations (for all the γ-variants within the PST) and the hard orientation by hard-mode dislocations. A PST crystal oriented in the soft orientation creeps significantly faster than the hard orientation [Figure 4.29]. The soft orientation, dominated by dislocations that glide parallel to the lamellar interface is unhindered by the interfaces and thus creep faster. Hard oriented grains, dominated by hard-mode
dislocations, i.e. those dislocations whose slip plane is NOT parallel to the interface plane, are much stronger due to the constraints offered by the interfaces.

A FL microstructure consists of colonies that are oriented in both soft and hard orientations. It is easy to perceive that immediately after loading and during primary creep, the soft oriented grains deform much more rapidly than the hard oriented grains. The large instantaneous strains in the FL alloy lends support to this observation [Figure 4.20 and 4.21]. In their extensive study, the Parthasarathy et al. [13] calculate the primary strain accumulated in the polycrystals and the PST crystals [Figure 4.30]. The primary creep strain data suggests that primary creep behavior of polycrystals and soft-oriented PST crystals are very similar.

As deformation in the soft oriented grains proceeds, the hard oriented ones load up. With increasing strain, von-Mises compatibility criterion requires that both soft and hard oriented grains deform compatibly. This implies that rate of deformation of the soft colonies will be limited by the rate of accommodation in the hard oriented grains. Hence at strains corresponding to the minimum strain rate, it is probably the rate of deformation of the hard oriented grains which controls the overall rate of deformation, especially keeping in mind the fact that most grains are probably NOT favorably oriented for soft-mode slip. Load-shedding models of creep and FEM models [14] of the behavior of polycrystalline FL alloys predict a similar response: softer grains controls the behavior initially, while harder ones control the steady state behavior.

Careful creep studies on oriented PST crystals and FL crystals have shown that the steady state creep behavior of FL crystals appears to imitate both that of the soft oriented and the hard oriented PST's at different stresses [Figure 4.31][15]. At high
stresses, the creep rates and stress exponents closely parallel that of the hard-oriented PST crystal, while the low-stress exponent is close to that of the soft oriented PST. By the analysis of results from our backflow tests, the following conclusions can be drawn:

a) The back-stress in the PST alloy is significantly larger in the hard orientation. Since there are no grain boundaries in the PST alloy, this has to be a lamellar spacing effect. In support, the smaller backstresses in the soft orientation indicates that there is no lamellar spacing effect (as expected). Back stress in the hard-oriented lamellar grains has already been suggested to result from channeling stress [Section 4.3.1]. Reducing the lamellar spacing should thus, increase the back stress. The origin of backflow is shown schematically in Figure 4.32a. During forward flow, the hard-mode screw dislocations lay out segments along the interface. On unloading, these near-edge segments will attempt to minimize line energy by zipping back. If the laths are too narrow to have jogs, the unzipping process is rapid. However, if the hard-mode dislocation is pinned by jogs, as shown in Figure 4.32b, the unzipping process is hindered in the backward direction (due to the presence of jogs). This mechanism also explains the observation that the backflow behavior is very protracted in the FL alloy and the hard-oriented PST crystals (backflow in the equiaxed and the 45° PST, does not last very long).

b) The backstresses in the FL alloy is initially small but increases rapidly to a significantly larger value. It can be interpreted that the lamellar spacing has little effect early on, but it has a substantial effect at and beyond minimum creep. This strengthens our view that creep in FL alloys is controlled by soft grains initially and by hard grains at the steady state.
c) The lack of lamellae explains the small back stresses observed in the equiaxed alloy (compared to FL). The equiaxed alloy and the FL alloy show an increase in the backstress with strain, underlining the role of compatibility between grains in polycrystalline materials. Plastic anisotropy between grains inherent to the FL material leads to the steeper increase in backstress with strain. In a creep curve this translates into a strong, exhausting primary transient for FL alloys. This has been observed by several researchers [16, 17]

In apparent contradiction, work by Parthasarathy et al [13] suggests that steady-state creep deformation in FL alloys is dominated by the soft-oriented grains (Figure 4.33). Their claim is based on the minimum strain rate versus stress data for variously oriented PST and FL polycrystals. However, this behavior is contradictory to the room temperature yield behavior where significant load shedding is observed with increasing strain. It has been suggested, that at room temperature the polycrystal behaves like the soft-oriented PST at micro-strains, but work-hardens rapidly to reach the flow stress of the hard oriented 0 degree PST crystal before 0.2% strain is reached [11]. Apart from this anomaly, there is another serious weakness to the claim made by Parthasarathy et al [13]. If the creep behavior of the polycrystal were indeed controlled by "soft-oriented" grains, then there should be little role of lamellar spacing (soft-oriented grains are dominated by soft-mode dislocations which move parallel to the lamellar interface). However, there is abundant evidence that finer lamellar spacing leads to significant creep strengthening. The authors suggest that there might be a relationship between domain spacing and lamellar spacing. The other probable cause, they suggest, is the change in the composition of gamma with varying lamellar spacings. These hypotheses are not verified
and thus appear weak (especially in view of the work by Maruyama et al [4-6], who show that creep rates are unaffected by $\alpha_2$ volume fraction, and hence the $\alpha_2$ and gamma composition)

The data presented by Parthsarathy et al, can however be re-interpreted. A closer look at their data indicates that the polycrystal was tested in the stress range of 100-230 MPa. They report a stress exponent of 3-4. These observations suggest that their data set probably lies in the low-stress regime. This also hints that soft-oriented grains dominate low-stress creep. The observation that soft oriented PST crystals and polycrystals have similar stress exponents at low stresses only goes to strengthen this hypothesis [Figure 4.31]. (The low stress regime will be discussed in detail in Section 4.6)

4.5. Model for predicting creep rates in FL microstructure

In this section we will develop a suitable scheme for accounting for the distribution of lamellar spacing. As shown in section 4.3.1, there exists a backstress in lamellar microstructure that is related to the stress required for channeling ($\tau_b = E_{dipole}/\lambda b$). When $\tau < \tau_b$, then there is no effective stress. The fully lamellar structure has a distribution of lamellar thickness (see Figure 4.34), and so for a given applied stress, lamellae thinner than a critical cut off $\lambda_c$ would not experience any effective stress and would not participate in the deformation process. This is the basic premise for an approach to modeling creep rates in fully lamellar structures with a distribution of lamellar spacing. This assumption seems to agree with microstructural observations of low dislocation activity in the thinner lamellae where dislocation multiplication appears to be limited. As the applied stress increases, the critical limit $\lambda_c$ decreases and a greater
volume fraction of the material is able to participate in the deformation process. Additionally, if the lamellar spacing is wider, a greater volume fraction of the material participates in the deformation process [Figure 4.34].

Since the strain rate in each lamella is different, we need an averaging scheme, which can best represent the effect of a lamellar spacing distribution. As shown in the previous section, the behavior of the FL material most closely parallels that of the N oriented PST crystal, hence we will average the creep rate assuming that the minimum creep rate for the FL alloys is the same as that for the N orientation. As shown in Figure 4.35, this is an isostress condition.

Note: For the sake of completeness, we will briefly explore an isostrain averaging approach in Appendix 3. This is assuming that the A-oriented PST crystal (the other hard orientation), is rate controlling.

If \( F(\lambda) \) is the volume fraction of laths of a particular lamellar spacing \( \lambda \)

\[
\dot{\gamma} = \frac{\int_{\lambda_{\text{min}}}^{\lambda_{\text{max}}} F(\lambda) \cdot v(\lambda) \cdot \rho(\lambda) \cdot b \cdot d\lambda}{\int_{0}^{\lambda_{\text{max}}} F(\lambda) \cdot d\lambda}
\]

4.15

\( v(\lambda) \) is the velocity of dislocations under the effective stress and \( \rho(\lambda) \) is the dislocation density as a function of the lamellar spacing (as shown in Section 4.3.2). Again, we can go from shear to tensile values of stress and strain-rate using \( \dot{\epsilon} = \dot{\gamma} / 2 \) and \( \tau = \sigma / 2 \) In order to proceed, the only other term that needs to be evaluated is the lamellar width distribution \( F(\lambda) \).

Measurements taken from TEM observations have been used to compile histograms of \( \gamma \) lath spacings for the Ti-48Al binary alloys in two lamellar spacings and
the K5 Alloy. These were done by tilting the sample so as to get the lamellar interfaces edge-on. A line intercept method, involving measurement of lamellar widths along a line perpendicular to the lamellar interfaces, was used for measuring the lamellar thickness distribution. A log normal fit seems to be suitable for number distribution \( N(\lambda) \):

\[
N(\lambda) = a \cdot \exp\left[ -\left( \frac{\lambda}{b} - c \right)^2 l(d^2) \right]
\]

where \( a, b, c \) and \( d \) are fitting parameters. The fit typically has a correlation close to 0.9. The log normal fit and the parameters for the two alloys are shown in Figure 4.36. The volume fraction distribution is calculated as:

\[
F(\lambda) = \lambda \cdot N(\lambda)
\]

Before attempting to predict the isostress-averaged strain rate using equation (4.17), we will describe the several assumptions made in the model:

- In this refined model, the lower cutoff is assumed as the critical cut off \( \lambda_c \): the width below which no effective stress is experienced. The upper limit is set to the largest lath measured in the distribution.

- The model also innately assumes that all laths have jogged dislocations and the rate-limiting process is the non-conservative motion of hard mode dislocation. It is possible that there are laths that might experience an effective stress but are not wide enough to develop jogs (jog spacing is greater than the lath spacing). This scenario is not considered.

- Strain contribution from soft mode dislocations is not considered for reasons given in the previous section (trans-lamellar soft-mode dislocations have been observed to
have jogs. However, these jogs may not have tall enough to make this process rate limiting, owing to the smaller mean free path).

- The jog spacing and the critical jog height in the velocity law have been set to be functions of the effective stress, rather than the applied stress. The value of $\beta$ used is 1.

The model predictions are shown in Figure 4.37 for the binary $\gamma$-TiAl alloy (two lamellar spacings). The data and predictions for the equiaxed microstructure are also indicated. This model predicts lower creep rates and higher stress exponents for FL structures when compared to the EQ structure. The actual creep rates predicted are slightly higher than the observed, but within reasonable error. The stress exponents predicted are also very similar to that observed in the high stress regime (~8). It also seems to reproduce the general trends associated with the lamellar spacing distribution. It predicts lower creep rates and higher stress exponents for narrow lamellar distributions. However, the model predicts the behavior only in the high stress regime accurately.

Attempt has been made to extend the model to the K5 alloy as well. The lamellar spacing distribution for the K5 alloy is shown in Figure 4.38. The dislocation density and jog spacing dependence of the binary alloy has been applied to the K5 alloy. The predictions are shown in Figure 4.39. The match is not as good as it is for the binary alloy. The predicted strain rates are slightly higher and the predicted stress exponent is significantly larger than the experimentally observed value. One source of this inaccuracy could arise from the fact that the representation of the lamellar distribution is not very accurate. The K5 alloy exhibits a bimodal type distribution with a large number of very fine lamellae, that are organized together in “packets” and lamellae that are relatively
coarser. This could easily skew the log-normal fit (the correlation coefficient R is smaller for K5 than for the binary alloys)

Minimum creep rate data obtained from Ott [18], for equiaxed Ti-48-2Cr-2Nb alloy is compared with the equiaxed binary alloy, for the same temperature (Figure 4.40). It is evident that the minimum creep rate for the Ti-48-2Cr-2Nb (Eq) alloy is significantly lower that that of the binary alloy. In both cases, the stress exponents are lower than for the lamellar structures and microstructural evidence clearly shows cusped screw dislocations. This strongly points towards a solute strengthening mechanism (complementing the effect of jogs), in the case of Ti-48-2Cr-2Nb. The K5 alloy is very similar in composition to the Ti-48-2Cr-2Nb alloy (with additional 0.2% W). The model in its present state accounts for the effect of lamellar spacing only and ignores possible solute effects. A consideration of these probable solute effects might lower the creep rates in Figure 4.39, and bring it closer to the actual experimental data. The manifestation of solute strengthening in the context of the model is not presently clear.

The isostress model predicts larger stress-exponent for narrower spacings, as is clear from the predictions for the K5 alloy. However it is possible that we are not probing the true "high stress" regime, but a transition regime between high and low stresses. Since the jogged-screw mechanism becomes difficult for these narrow spacings, the low stress mechanism will actually extend out to larger stress levels. Particularly if the low stress mechanism is diffusional creep (will be discussed in detail in the next section), narrower spacings could actually increase creep rates and favor the low stress mechanism even at larger stresses.
This isostress approach does, however provide an explanation for both the reduced strain rates and higher stress exponents observed for the lamellar structures. Note that this model cannot account for the significantly lower stress exponents observed at lower stresses. The possible mechanisms in the low stress regime are discussed in the next section.

4.6. Probable low stress mechanism

It has been previously suggested [19-21] that the low stress creep behavior is controlled by the movement of dislocations lying on the lamellar interface leading to interface sliding. It has been shown that the fault energies are reduced by almost half at two out of the three $\gamma/\gamma$ interfaces, making the $1/2[110]$ dislocation on the interface much more mobile than its counterpart in the bulk [22]. Direct evidence for the sliding of the $\gamma/\gamma$ interfaces has also been provided. We will explore the possibility of interface sliding as a probable creep mechanism

Just as at high stresses, at low stresses the soft-oriented grains will start deforming initially. Even at low stresses (where the strain rates are small and the compatibility issues may be less severe) von-Mises compatibility criterion will require that the hard oriented grains also deform with increasing strains. This deformation of the hard-oriented grains will however, not be via the "jogged-screw" dislocation mechanism, since it is extremely difficult to generate "hard-mode" dislocations (again due to channeling stress) in the hard oriented grains. However, the hard-oriented grains can deform purely by diffusional creep. This does not require the generation of dislocation into narrow channels. This suggests that at low stresses, diffusional creep controls the steady-state
creep rate. We can estimate the creep rates due to interface sliding based on the work by Raj and Ashby [23]. They have predicted the rate of diffusional creep for a microstructure consisting of different grain morphology (including lamellar). In essence, the FL microstructure can be assumed to consist of several parallel lamellar grains with the lamellar interfaces and the domain boundaries acting as short-circuit diffusion paths [See Figure 4.41]. This model necessarily requires that the interfaces slide (intrinsic, or Lifshitz sliding)[23]. Though the original model is a continuum model, this sliding in reality requires the movement of dislocations along the lamellar interface for folds or voids not to form at triple points [12]. This is viable since the motion of interfacial dislocations is quite easy in \( \gamma \) TiAl [22]. The creep rate predicted by the model is indicated below:

\[
\dot{\gamma}(\lambda) = 16 \cdot \tau \cdot \frac{\Omega}{kT} \cdot \frac{1}{l^2} \left( \frac{D_p}{R} + \frac{\delta \cdot D_B}{l \cdot R^2} \right)
\]  

(4.18)

where \( D_p \) is the interface/domain boundary diffusion coefficient and \( \delta \) is the interface width. If \( d \) is the inter domain boundary distance, \( R \) is the aspect ratio of lamellar grains = \( d/\lambda \) and \( l \) is the average grain size, which in this case is approximated as \( (\lambda d)^{1/2} \). The interdomain boundary distance is assumed to be 10 \( \mu \)m and the interface width is set at 2 Burger's vectors. The only unknown term is the boundary diffusion coefficient. No work has been done on evaluating the rate of lamellar (\( \gamma/\gamma \)) interface diffusion in \( \gamma \)-TiAl. However, there is an indirect way to estimate the diffusion coefficient of a boundary using the Borisov-Golikov relationship, as shown by Minkwitz et al [24]. The ratio of boundary diffusion coefficient to that of lattice diffusion is given by:
\[
\frac{D_B}{D_s} = \exp\left(\frac{2 \cdot \Omega \cdot E_B}{\delta \cdot k \cdot T}\right) \quad (4.19)
\]

\(\Omega\) is the atomic volume and \(E_B\) is the energy of the interface. The authors have used this relation to show that the diffusion coefficient on a coherent twin boundary is very small. Using the knowledge of the true-twin, 120° rotational and the pseudo-twin boundary, we can get an estimate of the interface diffusion coefficient. Using atomistic simulations, Fu et al have estimated the interfacial energies to be between 60 mJ/m² (for a true-twin boundary) to 270 mJ/m² (the other two \(\gamma/\gamma\) variants) [22]. Since most boundaries have been found to be of the true-twin type [25], the average value has been picked to be 100 mJ/m² (this has very little effect on the values predicted, as will be shown later).

We again assume that \(\dot{\epsilon} = \dot{\gamma}/2\) and \(\tau = \sigma/2\) and employ an isostress averaging, as shown:

\[
\dot{\gamma} = \frac{\int_{\lambda_0}^{\lambda_{\max}} F(\lambda) \cdot \dot{\gamma}(\lambda) \cdot d\lambda}{\int_{\lambda_0}^{\lambda_{\max}} F(\lambda) \cdot d\lambda} \quad (4.20)
\]

Combining equations (4.17),(4.18) and (4.20), we can predict the low stress behavior (shown alongside the high stress predictions) as shown in Figures 4.42 through 4.44. Since the low and high stress mechanisms are independent creep mechanisms, the creep rates are additive Assuming this, we can predict the creep behavior for the entire range of stresses, as shown in Figures 4.45 through 4.47. For a rudimentary model, its predictions appear respectable (especially for the fine lamellar material). Even though the model predicts creep rates in the ballpark of the observed values, there are concerns, which need to be addressed.
• Activation energy for an intermediate temperature diffusional creep process is expected to be less than that for lattice diffusion (in the case of grain boundary diffusion, roughly half). However our data (Figure 4.5) suggests that the activation energy probably corresponds to lattice diffusion. There is an explanation for these experimental values. As shown earlier, it is possible to estimate the ratio of grain boundary diffusion coefficient to that of lattice diffusion. The relative contribution of grain boundary diffusion and lattice diffusion can be evaluated by comparing the ratio of $D_B \delta$ to $D_S l$. If this value is small, then the contribution of grain boundary distribution is minimal. For a lamella of 200nm width, this ratio is approximately $5 \times 10^{-4}$ (Similar values have been suggested by Divinski et al. [26]). This suggests that the role of boundary diffusion at these temperatures is minimum and the diffusional creep mechanism is Nabarro-Herring creep (i.e. accounting for the lamellar grains). Hence the activation energy corresponding to lattice diffusion.

• The model predicts a linear dependence of strain-rate on stress. However, stress exponents of 2-3 are more often reported in the literature for the low stress regime [1-3]. The reason for the apparent stress exponent of 2-3 can be found in the techniques employed for measuring the stress exponents. Since the creep rates are very small (often as small as $10^{-9}$/s), researchers use stress-jump tests instead of monotonic tests for extracting the stress exponents. As is often the case with these tests, the strain rate that is reported is often not the "steady-state" rate but rather that at some stage of primary creep. The rate at which strain rate drops during primary creep is very small (since the creep rate itself is small) and this makes the determination of a "true" steady state elusive. So, it appears that the stress exponent reported in the literature at
low stresses probably hints more at the primary creep mechanism rather than during steady-state. Since the contribution of the soft-oriented grains to primary creep is large, the stress-exponent at low stresses is similar to that of soft-oriented PST crystals (3-4). There could be another explanation for the reported stress-exponent to be 2-3 instead of unity. In lamellar microstructures that have a coarser lamellar spacing, there is a fair chance a few lamellae are wide enough to allow dislocation generation, even at small stresses. Thus diffusional and dislocation creep can work in tandem, giving rise to intermediate stress exponents. This would suggest that a finer lamellar microstructure (one which minimizes dislocation creep), would have an exponent closer to unity. Our creep data suggests exactly this (Figure 4.45 through 4.47). The K5 alloy with the finest lamellar spacing has a low stress, stress-exponent of 1 while the coarse lamellar binary alloy has a stress exponent of 3. The fine binary with intermediate lamellar spacing has a stress exponent closer to 2.

- The model also predicts that the strengthening effect of lamellar spacing will be lost in the low stress regime. In fact, the model predicts the creep rates for the finer-spaced FL alloys to be higher (as is true with all diffusional creep mechanisms). There is some evidence in the literature indicating that the effect of lamellar spacing does indeed vanish at low stresses [4, 5, 27]. The strengthening effect indeed appears to vanish at low stresses as seen in Figure 1.14. There is also some evidence that the creep rates actually go up when the lamellar spacing becomes extremely small [28]. However, this is not supported by our data for FL binary Ti-48Al. Comparison between the K5 alloy and the Ti-48Al alloy at low and high stresses indicates that the effect of lamellar spacing does indeed vanish at low stresses for the K5 alloy.
However, we have to keep in mind that the two alloys are quite different in composition and any comparison will have to be taken with a pinch of salt. (Note: In Figure 1.14., the differences in creep rates between different alloys is much larger at high stresses than at low stresses)

We can speculate on some possible reasons for our observations. The lamellar spacing effect is not lost at low stresses probably because, some of the accommodation between the grains is still achieved via hard-mode dislocation in the hard oriented grains and lamellar spacing has an inverse effect on the velocity of hard mode dislocations. It is also possible that during primary creep, slip in the soft oriented grains takes place parallel to the interface, but via dislocations that glide across the lamellae (instead of parallel to the interface). These dislocations can pile up against the lamellar interface. This pile-up model leads to the observed lamellar spacing effect on the creep rate (even at low stresses). It should however be noted (see Table 4.1) that the so-called "true" soft mode deformation has the highest Schmidt factor and so the contribution from trans-lamellar "soft mode" may not be significant.

4.8 Conclusion and unresolved issues

Jogged 1/2[110] screw dislocations dominate the deformation microstructure in FL samples. The dislocation activity is inhomogeneous with extensive activity in thicker lamellae and little or no activity in thinner lamellae. It is proposed that there exists a critical lamellar thickness, which is related to the minimum stress required to cause channeling of dislocations: lamellae thinner than the cutoff thickness experience no
effective stress. The presence of back stresses is confirmed by back flow experiments. Additionally, it appears that primary creep is controlled by soft-mode deformation while the hard mode deformation determines the steady state creep rate. A refined jogged-screw velocity law for the FL structures has been developed. An isostress averaging scheme has been adopted in view of the fact that the FL material creep behavior most resembles that of the N-oriented PST sample. This isostress model predicts higher stress exponents and lower creep rates for finer lamellar distributions, in accord with observations.

It is evident that there are several unanswered questions especially in the low stress regime. One study indicates that the strain contribution via sliding is minimal. However, the techniques employed are questionable. Also, interfacial sliding will be prevalent only in alloys with relatively fine lamellar spacing. The non-existence of two stress regimes has been shown for lamellar Ti-48Al-2Cr-2Nb and this seems to be related to the fact that these alloys have a much wider lamellar spacing which allow dislocation creep processes even at low stresses. Additional TEM study is necessary in order to determine whether an actual change in the principal deformation mechanisms is responsible for the markedly different stress dependence observed in this regime.
LIST OF REFERENCES


22. C. L. Fu and M. H. Yoo, Scripta Materialia 37, 1453-1459 (1997).


Figure 4.1. Typical creep curves for the binary Ti-48Al (FL) alloy with two different lamellar spacings.

Figure 4.2. Creep curve of the K5 alloy at 760 C and 315 MPa.
Figure 4.3. Consistency of creep results between different samples of the K5 alloy tested under identical conditions.

Figure 4.4. The creep curve for the binary alloy at two different temperatures.
Figure 4.5. The minimum strain rate as a function of temperature compared for different alloys and a range of stresses.

Figure 4.6. The minimum strain rate as a function of stress of the various FL alloys and the equiaxed alloy. Lower creep rates and the existence of two stress regimes are characteristics of the FL microstructure.
Figure 4.7. Subgrain formation seen only at large strains and is restricted to the widest laths. K5 alloy crept at 760°C, 315 MPa to 4.7%.

Figure 4.8. Inhomogeneous deformation microstructure, with smaller dislocation densities in the thinner laths and significantly larger densities in the thicker laths. Binary lamellar alloy crept at 768°C and 207 MPa.
Figure 4.9. The presence of hard mode jogged screw dislocations in the binary lamellar alloy crept at 768C and 207 MPa is noted.

Figure 4.10. The presence of trans-lamellar soft-mode dislocations that are jogged (binary lamellar alloy crept at 768C and 207 MPa)
Figure 4.11. BF image (g=(111)) of K5SC unaged sample crept at 315MPa, 815°C, 0.41% strain (400s). Most dislocations are hard mode ordinary dislocations. Dislocation activity much higher in the wider laths. Several dislocations are jogged.

Figure 4.12. The jogged dislocation in crept K5 alloy, imaged with a) g = (111) and b) g = (002). Invisibility in case b indicates that the dislocation is an ordinary dislocation. Also note the prismatic loop trailed behind the jog.
Figure 4.13. Different beam directions used to image the [110](1\bar{1}1) jogged screw dislocation, a) $B = [\bar{1}10]$, b) $B = [201]$ and c) [311]. Case b and c are oriented at ~40° and ~58° to the glide plane along the screw direction. The increase in separation between the segments attached to the jog with increase in tilting proves that this is a tall jog. A rough estimate of the jog height is 57 nm.

Figure 4.14. The same two channeling dislocations viewed along two different beam directions. a) $B = [\bar{1}10]$, $g = (111)$ and b) $B = [2\bar{1}1]$, $g = (1\bar{1}1)$. Tilting the sample parallel to the lamellar interface shows that channeling dislocations lays out dislocation segments along the interface, as indicated.
Figure 4.15. A schematic of the jog-screw mechanism in lamellar microstructures. A Thompson’s tetrahedron indicated in the bottom as a reference.

Figure 4.16. A schematic of the jog-screw dislocation motion looking down at the glide plane.
Figure 4.17. The variation of the effective stress ($\tau_{\text{eff}} = \tau - \tau_b$) as a function of lamellar spacing.

Figure 4.18. TEM measurements of the dislocation density as a function of lamellar spacing.
Figure 4.19. A schematic showing the various parameters measured in a typical stress drop test.

Figure 4.20. The variation in instantaneous strain during a stress drop for the different microstructures. Note the large strains for the FL microstructure. Also indicated is the elastic strain during the stress drop calculated from a Young’s modulus of 153 GPa.
Figure 4.21. The variation instantaneous strain during a stress jump for the different microstructures. The strains for the FL microstructure is again the largest.

Figure 4.22. The ratio $M'$ for the different microstructures. The value of $M'$ is less than 1 for all cases except one.
Figure 4.23. The ratio $R'$ for the three PST orientations. The N orientation has an $R'$ value almost an order of magnitude higher than the other two.

Figure 4.24. The ratio $R'$ for the FL and equiaxed microstructures. FL microstructure has an $R'$ value, an order of magnitude larger than that for the equiaxed indicating large back stresses.
Figure 4.25. The ratio $R'$ for the FL is compared with that of the PSTs. FL microstructure has an $R'$ value comparable to the N-orientation at strains corresponding to minimum strain rate.

Figure 4.26. The anelastic strain for the three PST orientations compared. Trends are similar to that observed for $R'$. 
Figure 4.27. The anelastic strain for the equiaxed and the FL microstructure is compared. Trends are similar to that observed for R’.

Figure 4.28. The anelastic strain for the FL microstructure compared with that for the three PST orientations. Trends are similar to that observed for R’.
Figure 4.29. Anisotropy in the creep behavior of the PST crystal is evident. The B orientation creeps significantly faster than the two hard orientations.

Figure 4.30. Creep data from Parthasarathy et al comparing the primary creep strains of the various microstructures. The FL polycrystal has primary strains comparable to the 45° oriented PST [13].
Figure 4.31. Data from Wegmann et al. suggests that at high stresses, the creep rates and stress exponents of the FL polycrystal closely parallels that of the hard-oriented PST crystal, while the low-stress exponent is close to that of the soft oriented PST [15].

Figure 4.32a. The origin of backflow driven by the unzipping of the dislocation segments along the interface.
Figure 4.32b. The backflow process is hindered when the channeling dislocation has jogs. This leads to a prolonged backflow behavior.

Figure 4.33. Creep data from Parthasarathy et al. suggesting that the FL polycrystal behaves like the 45° oriented PST [13]
Figure 4.34. The premise for modeling creep rates in fully lamellar structures with a distribution of lamellar spacing is that lamellae thinner than a critical cut off $\lambda_c$ would not experience any effective stress and would not participate in the deformation process.

Figure 4.35. Schematic of the isostress averaging approach.
Figure 4.36. The log normal fit for the fine and coarse binary lamellar alloy. The fit parameters are indicated.

Figure 4.37. The model predictions are indicated along with the experimental values. Also shown are the experimental values and model predictions for the equiaxed microstructure. The model predicts the general trends of FL alloys, i.e., higher stress exponents, lower creep rates (compared to equiaxed) and the lamellar spacing effect.
Figure 4.38. The log normal fit and the fit parameters for the K5 alloy are indicated. The fit is much poorer for K5 alloys.

Figure 4.39. The model predictions and the experimental values for the K5 alloy are shown.
Figure 4.40. Data from Ott, suggest that there is an effect of alloying additions on the creep behavior of equiaxed microstructures. Solute effects have been ignored in our model [18].

Figure 4.41. Schematic of the interface sliding model suggested by Raj and Ashby adapted for lamellar microstructures (adapted from ref [23])
Figure 4.42. The low and high stress predictions for the coarse lamellar binary alloy

Figure 4.43. The low and high stress predictions for the fine lamellar binary alloy
Figure 4.44. The low and high stress predictions for the K5 alloy

Figure 4.45a. The cumulative strain rate for the entire stress regime for the coarse lamellar alloy (assuming interface sliding and jogged-screw mechanism to be independent processes)
Figure 4.45b. The cumulative strain rate for the entire stress regime for the fine lamellar alloy

Figure 4.45c. The cumulative strain rate for the entire stress regime for the K5 alloy
Table 4.1. Schmid factor for each slip system (for ordinary slip) for a PST crystal oriented in the A (0°), B (45°) and N (90°) orientations (This calculation was made assuming that the Burger's vector is oriented at 45° to the stress axis). The Schmid factor is shown for all six γ variants. The mode of slip (hard or soft) is also indicated. The darker shade of grey indicates the slip-system with the maximum Schmid factor. The lighter shade of grey is the slip system with the second highest Schmid factor. It is evident that for the soft orientation, true soft mode has the maximum Schmid factor in all variants but one. For the hard orientations, hard-mode deformation is strongly favored in most variants.
CHAPTER 5. MICROSTRUCTURAL STABILITY OF FULLY LAMELLAR ALLOYS UNDER CREEP

From the previous chapter it is obvious that the creep properties of the FL alloys are far superior to both duplex and equiaxed microstructures. It also appears that control of lamellar spacing is key to the development of alloys for high temperature applications. Since the creep properties of these alloys are closely tied to the underlying structure, it is also important to understand the effect of high temperature exposure on the microstructural stability of the FL TiAl alloys.

Many an early test on FL alloys was performed under tension. Failure of the material was often attributed to damage processes including cavitation. Only in the past five years has any attention been paid to the changes in the microstructure occurring during creep. Though these changes could lead to accumulating damage and eventual failure in tensile tests, these could lead to extended periods of tertiary creep under compression. The aim of this chapter is to delve into issues related to microstructural stability of FL alloys under exposure to temperature and stress and its effect of the creep behavior.
5.1 Effect of temperature.

Figure 5.1. shows the TEM micrograph of FL K5 alloy in the as-solutionised condition (the term "unaged" will also be used for this condition). Centered dark field images using $\alpha_2$ reflections were used to image the $\alpha_2$ laths. Lamellar spacing measurement was done by tilting the sample such that the lamellar interfaces are edge-on. $\alpha_2$ laths are seen to be uniformly distributed in the unaged K5 material, with numerous $\alpha_2$-$\gamma$ boundaries. The material was given a 24hr/900°C heat treatment in air (aging treatment). TEM micrographs indicate that there is substantial thinning of $\alpha_2$ lamellae after aging (Figure 5.2). Distribution of $\gamma$ and $\alpha_2$ lath spacings before and after aging treatments (Figure 5.3 and Figure 5.4) confirm the loss of $\alpha_2$ during aging. The volume fraction of the $\alpha_2$ phase was measured to be 22 vol% in the unaged condition. The volume fraction of $\alpha_2$ phase drops to 7.2 vol%. There is an increase in the number of $\gamma$-$\gamma$ interfaces due the complete dissolution of $\alpha_2$ between two adjacent $\gamma$-laths. There also appears to be a significant increase in the number of ultrafine gamma laths that are arranged in packets (Figure 5.5), which is also apparent from the $\gamma$ lath distribution after aging (Figure 5.4). This packet structure is rarely observed in the unaged condition. The observation of plate like gamma nuclei within the $\alpha_2$ phase (Figure 5.5) seems to indicate that the ultrafine packet structure is formed by the nucleation and growth of secondary $\gamma$ plates within $\alpha_2$. Also observed in the K5 material is the precipitation of B2 phase that is often seen in dissolving $\alpha_2$ laths (Figure 5.6). EDS measurements confirm that these precipitates are rich in Cr, Nb and W.
Figure 5.7 shows the TEM micrograph of the as-solutionised K5SC alloy. The K5SC alloy contains carbon and silicon and its processing is similar to that of the K5 alloy. Prior to aging, the $\alpha_2$ content is greater than 20 vol% and the distribution is similar to that of the K5 alloy. There are no precipitates. After a 24hr/900°C aging treatment, reduction in the thickness and distribution of $\alpha_2$ laths is observed, in this case as well [Figure 5.8]. The $\alpha_2$ volume fraction decreases to 8-vol%. The formation of ultra-fine $\gamma$ is observed in this case as well.

Such a drop in the $\alpha_2$ volume-fraction during aging is well-documented [1-5]. Fine lamellar structure with large volume fractions of $\alpha_2$, is a structure far from equilibrium and is thus not a stable structure [3]. For a binary alloy, it is evident from the binary phase diagram that retention of 20 vol% $\alpha_2$, corresponds to an $\alpha_2$ composition that is far from stoichiometry (close to 40% at.% Al instead of 25 at.% Al). In the K5 alloy W and Cr very weakly partition to the $\alpha_2$ phase [5]. However, their role in stabilizing retained $\alpha_2$ appears to be minimal (the abundant formation of B2 particles stabilized by Nb, Cr and W, on aging provides additional support). It is evident that the retention of metastable $\alpha_2$ prior to aging provides a large driving force for the dissolution of $\alpha_2$ during aging. Both K5 and K5SC samples have similar $\alpha_2$ volume fractions prior to and after aging. The fact that both K5 and K5SC samples retain 6-8 vol% $\alpha_2$ after aging suggests that this is the equilibrium volume fraction (the presence of Si and C does not seem to affect the amount of $\alpha_2$ finally retained).

As seen in Figure 5.5, aging leads to the precipitation of B2 particles in the K5 alloy. In the K5SC material, aging leads to the aligned precipitation of carbides and
silicides, which decorate the interlamellar boundaries and dissolving \( \alpha_2 \) laths [Figure 5.9]. Similar equilibrium \( \alpha_2 \) volume fraction in K5 and K5SC samples suggests that the \( \alpha_2 \) dissolution process precedes the precipitation process: the growth of the \( \gamma \) phase at the expense of the \( \alpha_2 \) phase leaves the precipitates along the \( \alpha_2/\gamma \) interface. It is likely that as the \( \gamma \) phase is formed, C and Si get enriched in the remaining \( \alpha_2 \) (these alloying additions tend to partition preferentially to \( \alpha_2 \)) and when the \( \alpha_2 \) phase has reached maximum C and Si solubility, they are rejected into the \( \gamma/\alpha_2 \) interface to form precipitates [6, 7]. B2 precipitates are not seen frequently in K5SC. This could be because silicides have been known to precipitate preferentially from pre-existing B2 precipitates, rather than directly from dissolving \( \alpha_2 \) [10]. Silicide precipitation appears to be at the cost of dissolving B2.

Two types of carbide precipitates have been characterized in alloys containing carbon. Below the aging temperature of 800°C, many researchers [8-11] have observed a uniform distribution of fine precipitates in the \( \gamma \)-phase. These precipitates (Ti\(_3\)AlC) have a perovskite type structure and are designated as P-type precipitate. These precipitates have a needle/rod like morphology and lie parallel to the [001] direction of the TiAl matrix. After prolonged aging, the precipitates become rounded cubes with sides nearly parallel to the [010] and [100] directions of the matrix. These precipitates are coherent and hence nucleate homogeneously within \( \gamma \) phase. However preferential precipitation occurring along dislocations has also been observed [9, 12]

By aging at temperatures above 800°C (or extended aging at low temperatures), plate type precipitates with hexagonal crystal structure have been observed [9] These are the Ti\(_2\)AlC-type precipitates and are designated the H-phase. The plates are parallel to the
\{111\} of the TiAl matrix. Since the aging temperatures employed in our experiments were all above 800°C, we expect most of the precipitates to be of the H-type. These precipitates are incoherent and so they precipitate on the \(\gamma-\alpha_2\) interface and never in the gamma matrix. Precipitation of H-type carbides on interface dislocations has also been proposed.

The silicide precipitate that is observed is of \(\zeta\) - type \((\text{Ti}_3\text{Si}_3)\) Owing to the significant solubility of Al in \(\text{Ti}_3\text{Si}_3\) and a more likely chemical formula is \(\text{Ti}_5(\text{Si},\text{Al})_3\). \(\text{Ti}_3\text{Si}_3\) precipitates have a D8\(_s\) crystal structure and a block or needle shaped morphology. These precipitates are incoherent with both \(\alpha_2\) and \(\gamma\) phases and so observed along the \(\gamma-\alpha_2\) interfaces [13]

5.2. Creep behavior of aged K5 and K5SC alloy

Figures 5.10 and 5.11 indicate the creep behavior of the K5 and the K5SC alloy in the aged condition at the same stress level. Both alloys have the same \(\alpha_2\) volume fraction and similar lamellar thickness distribution, with the only microstructural difference being the presence of the precipitates. The K5SC alloy with Si and C outperforms the K5 material by an order of magnitude in creep rate. It is evident that the addition of Si and C is very beneficial for the creep properties. Two views exist on the possible source of this effect. The first is via solute strengthening and the second is via precipitation strengthening.

It has been proposed by Worth et al, that the strengthening is achieved purely by solute strengthening [14]. This is based on their observation of stress dependence close to 3 and the relative independence of creep resistance on carbide spacing. However it must
be pointed out that their conclusions are based on equiaxed and duplex microstructures (and mostly P-type precipitates in the $\gamma$-phase).

The possibility of precipitate strengthening has been previously proposed [5, 9, 11, 15-17]. We concur that the main source of strengthening in C and Si containing alloys is precipitation hardening. TEM studies of crept microstructures of the K5SC alloy indicate extensive interaction between the precipitates and the dislocations at the interface (Figure 5.12.). These precipitates being incoherent, this interaction is of an attractive nature (Arzt-type).

The precipitates play a three-fold role towards improving creep properties (Figure 5.13). First, it prevents the motion of soft-mode dislocations along the interface. Dislocations in the lamellar interfaces are seen to be pinned by this regular array of precipitates. Edge on views of the interface shows a highly irregular and wavy lamellar interfaces in the K5SC alloy and smooth interfaces in the K5 alloy. Pinning of interfacial dislocations by interfacial precipitates (and the associated strain contrast) leads to the wavy interface in the K5SC alloy. In comparison, there is nothing interfering with the motion of interfacial dislocations in the K5 alloy and they seem to zip along the interface easily, hence the smooth interface. Slowing down deformation in the soft mode will lead to a smaller primary strain and eventually slower minimum creep rate (the loading up of hard mode dislocations is delayed).

Second, the interfaces are the primary source of dislocations within the $\gamma$ phase. The precipitates interfere with the operation of dislocation sources on the interface and the expansion of newly formed dislocation loops. Third, strings of precipitates are often found in the middle of $\gamma$ laths [Figure 5.14]. The formation of these precipitates can be
visualized to be by the complete dissolution of an $\alpha_2$ lath originally flanked on either side by the same $\gamma$ variant. These precipitates help in obstructing the motion of dislocations within the $\gamma$ laths and this is seen in the frequent pinning and bowing of dislocations at these precipitates. It is interesting to note that crept samples of K5SC have a significantly lower dislocation density within the thicker lamellae when compared to the K5 samples. (Note: Beddoes et al. suggest the possibility of precipitate strengthening due to B2 particles [18]. B2 precipitates formed in the K5 material are relatively large and their effect in precipitation strengthening appears to be minimal. Increase in B2 precipitation, its morphology and its effect on creep are issues that need to be studied further.)

To eliminate any possible solute strengthening effects, the creep behavior of K5SC (aged) material is compared with K5SC (unaged). The unaged material has no precipitates and has the Si and C retained in solid solution. The comparison shown in Figure 5.15, indicates that the aged condition is much more creep resistant, and so precipitation strengthening appears to dominate over solute strengthening effects (if any). Also indicated in Figure 5.15 is the creep behavior of the K5 (aged) alloy. Interestingly, the initial creep rate and the tertiary creep rate of the unaged K5SC alloy is larger than that of the aged K5 alloy. This is despite the fact that there is precipitation in the K5SC alloy during the course of creep. This trend may be due to the fact that the unaged K5SC still retains a non-equilibrium volume fraction of $\alpha_2$ which rapidly dissolves, especially during the initial stages of creep. This underlines the importance of stabilizing the microstructure prior to creep. The presence of $\alpha_2$ laths, the stronger second phase, has often been cited as the reason for the better creep properties of fully lamellar structures [18, 5]. A drop in room temperature yield strength has been associated with the removal
of the $\alpha_2$ lamellae. This study shows that removal of $\alpha_2$ prior to creep might not be detrimental, but rather beneficial, as evident from the fact that the unaged K5SC containing more $\alpha_2$ (~25%) is clearly weaker than the aged K5, which has less $\alpha_2$ (~8%) (both have no precipitates at the beginning of creep testing). This also calls to question the validity of existing "composite" models for creep in FL alloys. It appears that the stability of the microstructure may be more important for obtaining high creep strengths than the initial volume fractions of the constituent phases. Also to be noted from Figure 5.15 is the fact that all three alloys undergo a protracted tertiary creep (under compression). This behavior has also been observed for PST alloys tested under compression. With this in mind, the effect of stress and temperature on the creep will be characterized. Using the knowledge gained, probable primary, secondary and tertiary creep mechanisms will be suggested.

5.3. Microstructural stability during creep.

Several tests were devised to investigate the relative effect of stress and temperature on the stability of FL microstructures. The test material is the K5SC alloy in the unaged condition, since this is the one that exhibits worse properties than both the K5 aged alloy and the K5SC aged alloy at all stages of creep. In one set of tests, the K5SC alloy was exposed to the test temperature (815°C) for different periods of time. The test temperature was chosen to avoid the formation of the lower temperature carbides. These samples will be referred to as "exposed". In the second set of experiments, the samples were crept to different strains and for different times and at two different stress levels
(315 MPa and 150 MPa). These samples will be referred to as "crept". The entire matrix of experiments is indicated in Table 5.1.

Both exposed and crept samples were examined in a TEM and the evolution of microstructure with time, strain and stress was characterized.

5.3.1. Effect of exposure

The effect of temperature has been discussed in section 5.1 in detail. This study confirms the results of the aging studies (though the temperature here is lower). TEM micrographs 5.16 and 5.17 show the effect of temperature on the microstructure. With increasing exposure time, the total $\alpha_2$ volume fraction drops from 29% to 10% (Figure 5.18). The drop in the $\alpha_2$ volume fraction follows parabolic kinetics, as expected for a diffusion based phase transformation. The distribution of $\alpha_2$ thickness is indicated in Figure 5.19. Thinning of $\alpha_2$ laths is observed. Within the first few hours of exposure the average $\alpha_2$ spacing drops drastically (Figures 5.20). The effect on $\gamma$ spacing distribution is also very telling (Figure 5.21). Though the coarse lamellar spacings are not affected much by exposure, there is a definite increase in the number of very fine $\gamma$ lamellae. All this suggests that most of the $\alpha_2$ loss occurs by the nucleation of secondary $\gamma$ within the retained $\alpha_2$. Precipitates appear to form only after 16.5 hrs of exposure. It has been shown by Kim et al, that 12 hr aged samples do not fare as well as the 24 hr aged samples under creep conditions. It now appears that this could be related to the time required for the precipitation of observable carbides and silicides.
It is also observed that the dislocation density in the exposed samples is much higher than the unaged sample [Figure 5.22]. The high dislocation density in the matrix suggests that phase transformation by itself can lead to dislocation generation during aging. Most of these dislocations are hard-mode dislocations. This finding has important implications on the creep behavior. The possible mechanisms for the generation of hard-mode dislocations during exposure are suggested in Section 5.4. In the next section we will look at the combined effect of stress and temperature.

5.3.2. Effect of stress and exposure.

Figures 5.23 through 5.26 indicate the histograms of the $\alpha_2$ and $\gamma$ spacings as a function of exposure time and the applied stress. $\alpha_2$ dissolution and the formation of packets of ultrafine $\alpha_5$ and $\gamma$ laths are seen in Figures 5.27 and 5.28. Figures 5.29 and 5.30 indicate the change in $\alpha_2$ volume fraction as a function of exposure time and strain. It is clear that $\alpha_2$ is lost during creep conditions too. While Figure 5.29 indicates that the loss of $\alpha_2$ is faster at higher stresses, Figure 5.30 shows the opposite trend with strain. It is difficult to isolate the effects of time and strain, since a lower stress exposure leads to a smaller strain in the same amount of time.

In Figures 5.31 and 5.32, the $\alpha_2$ volume-fraction and spacing is indicated as a function of time for the crept and the exposed samples. It is noteworthy that loss of the $\alpha_2$ is apparently retarded under the application of stress. Even samples crept an order of magnitude longer time (at 150 MPa) have wider $\alpha_2$ laths than samples not exposed to stress, suggesting that the application of stress impedes the dissolution process. This is
counter-intuitive and is not supported by overwhelming evidence in the literature in favor of stress-induced transformation.

Microstructural observations also suggest that there is an abundance of equiaxed $\gamma$ grains (of up to 10 microns) which are related to the lamellar structure via low angle (5-15°) grain boundaries (Figure 5.33). Recrystallized “equiaxed” grains are seen only during creep (not during aging). The extent of recrystallization increases dramatically with increasing stress, strain and exposure time. Recrystallized $\gamma$ grains appear to form preferentially in the vicinity of $\alpha_2/\gamma$ packets. The volume fraction was characterized by a line-intercept technique on the centered dark field images using $\alpha_2$ reflections. Due to this the volume fraction and $\alpha_2$ spacing reported assumes the entire material to be lamellar and does not account for the recrystallized gamma grains. This explains the discrepancy in $\alpha_2$ volume fraction after creep. $\alpha_2$ is probably still lost to the same extent (or maybe greater) under the application of stress, but mostly in the form of recrystallized $\gamma$ grains rather than by the growth of primary gamma lamellae, or by the nucleation and growth of secondary gamma.

This observation prompts us to explore the possible driving force for dynamic recrystallization (recrystallization is rarely observed in crept samples that were pre-aged). As $\alpha_2$ transforms to $\gamma$, there is an overall increase in volume. This is because of the difference in density between $\gamma$ and $\alpha_2$. The total change in volume can be easily estimated assuming mass conservation during the transformation.

$$\Delta V = \frac{V_{f,initial}}{V_{f,final}} \cdot (R - 1) + 1$$ (5.1)
where $\Delta V$ is the fractional increase in volume, $V_f^{\text{initial}}$ and $V_f^{\text{final}}$ are the initial and final $\alpha_2$ volume fractions and $R$ is the ratio of the density of $\alpha_2$ to the density of $\gamma$. The room temperature density for stoichiometric $\alpha_2$ is 4.21339 g/cm$^3$ and that of stoichiometric $\gamma$ is 3.79993 g/cm$^3$, which gives a ratio $R$ of 1.10899. The coefficient of thermal expansion (CTE) of the two phases can be taken into account to calculate the actual density of the phases at the exposure temperature. Assuming that $\text{CTE}_{\alpha_2} = 12.5 \times 10^{-6}/\text{K}$ [19] and $\text{CTE}_{\gamma} = 13.0 \times 10^{-6}/\text{K}$ [20] (and assuming isotropic expansion), the ratio $R$ at the creep temperature (815°C) is 1.1533. A loss of 19% $\alpha_2$ (going from 29% to 10%) can lead to a total volume increase $\Delta V$ of 2.9%. This translates to a linear strain of almost 1%, which is substantial from a creep perspective.

It may be argued that the metastable $\alpha_2$ (with nearly 40%) has a significantly different density compared to stoichiometric $\alpha_2$. Making a simple assumption that increased Al concentration in the metastable $\alpha_2$ is due to Al$_{\text{Ti}}$ anti-sites and using the lattice parameter information as a function of Al content [21], it is possible to estimate the density of $\alpha_2$ as a function of composition. For a starting $\alpha_2$ composition of 40 at.% Al, the density is 4.015, which is still substantially different from that of $\gamma$ (this leads to at least 1.9% volume increase; a proper integration accounting for the increasing $\alpha_2$ density, with aging will predict a $\Delta V$ value between 1.9 and 2.9 %). The minor partitioning of Cr and W to the $\alpha_2$ phase simply exacerbates the density differential.

In the absence of stress, the dissolution process and the concomitant expansion is unhindered. Based on Le Chatelier’s principle, the application of a hydrostatic compression should therefore slow down the transformation process. The stress-state is
not hydrostatic under compressive creep. However, the application of stress will indeed bias the dissolution process such that transformation that causes volume increase parallel to the compressive loading axis is suppressed and transformation that cause volume increase perpendicular to the compressive loading axis is promoted.

It is thus crucial to know the direction in which the volume change occurs. If we look at the basal plane of Ti₃Al and the (111) plane of TiAl, which form the lamellar interface, it is seen that there are 3 Ti atoms to every Al atom in Ti₃Al and 1 Ti atom for every Al atom in TiAl [Figure 5.34]. With the knowledge of lattice parameters, it is easy to estimate the areal density of each layer. The difference in the areal density of the interface layers in the two phases is 12% (α₂ being denser). This accounts for a major part of the overall density differential (~11%). The distance between adjacent close packed layers is virtually identical in α₂ and γ, suggesting that the necessary change in the density between the two phases, can be achieved purely by the change in the density of the interface layers. The increase in volume accompanying the phase transformation is thus parallel to the lamellar interface. This hypothesis agrees with the observation of the ultrafine lamellar microstructure within α₂ laths. If volume increase were perpendicular to the interface, then the compressive stresses created by the formation of the several fine parallel gamma plates in the α₂ would be enormous (the local strains will be large in view of the ultrafine scale). Only a volume increase parallel to the interface can give rise to the ultrafine structure.

This suggests that loss of α₂ through a lamellar transformation is promoted in lamellar grains which are oriented with the lamellar interfaces perpendicular to the compressive axis, while it is suppressed in grains that are oriented with the interface
plane parallel to the compressive axis. The first type of grains will be called N-oriented and the second type will be called A-oriented (following the PST convention). In the N-oriented grains, the lamellar dissolution of $\alpha_2$ is greatly accelerated. In the A-oriented grains, transformation via the formation of equiaxed grains is preferred. The observation of recrystallized equiaxed grains within the $\alpha$ packets supports this view. This hypothesis still needs to be thoroughly tested. However, there is evidence indicating that there might be some verity to this idea.

Our TEM samples were prepared by sectioning the crept sample perpendicular to the compressive axis. While undertaking the quantification of $\alpha_2$ lath spacing and volume fraction, most grains picked were those, whose lamellar interface could be observed edge-on with minimal sample tilting in the TEM. Due to this almost all the data acquired is from A-oriented grains which are favorably oriented for recrystallization and not for “lamellar” loss of $\alpha_2$. Preliminary observations indicate that the extent of recrystallization wanes on going from grains of A-type to B-type. There is a clear need for quantification of the extent of recrystallization as a function of angle $\phi$.

The second and more conclusive evidence comes from the creep work on PST crystals by two independent groups. Wegmann et al. [22] report a lower creep rate for the N oriented PST crystals, but a significantly poorer tertiary creep behavior [Figure 5.35]. They do not speculate on the reason for this weakening. Lin et al. [23] have found the same behavior, but difference in the tertiary creep behavior between the two orientations is even more dramatic as seen in Figure 5.36. They observe an accentuated loss of $\alpha_2$ in the N orientation while the loss in the A orientation is minimal (though kinking of the $\alpha_2$
bands is observed). Finally our creep data for the N and A orientation suggests that though the N orientation exhibits the lower creep rate of the two hard orientations, it undergoes severe tertiary behavior, which is not observed in the A-oriented PST crystal [Figure 5.37]. PST crystals typically have ~ 15 vol.% $\alpha_2$ and can be expected to be unstable microstructurally. It is to be noted that all these tests were conducted in compression. Preliminary SEM observations suggest that the "lamellar dissolution is indeed accelerated in PST crystals crept in the N-orientation (3 % $\alpha_2$ finally retained), while it is retarded in the A-oriented samples (6 % $\alpha_2$ finally retained). There is virtually no creep data on the tensile creep behavior of PSTs. It will be interesting to see if the trends in the tertiary creep regime, reverse when going from compression to tension (A-orientation would have a worse tertiary creep behavior than the N orientation)

The recrystallized grains can actually grow at the expense of the lamellar grains and lead to its eventual disintegration. It is suggested that the formation of recrystallized $\gamma$ grains leads to the loss of the lamellar structure (and the accompanied loss of strengthening). Significant dislocation activity has been observed in the equiaxed recrystallized grains as shown in Figure 5.33 which may in part explain the more severe tertiary creep behavior in the unaged K5SC alloy.

5.4. **Effect of aging on primary creep**

It was assumed in Chapter 4 that the primary creep behavior is controlled by grains oriented for soft mode deformation. This involves the glide of dislocations parallel to and preferentially along the interface. The lower primary creep rates for the K5SC aged material (due to the interaction of precipitates and interfacial dislocations) validates
this hypothesis. The lack of precipitates in the K5SC unaged material and the K5 aged material accounts for the large primary creep rates in those alloys.

The transformation from \( \alpha_2 \) to \( \gamma \) can take place by the passage of Shockley partials on alternate close packed planes. The movement of Shockley partials along the interface can be expected to be heightened in the unaged alloy, since there is the extra driving force for transformation. The increase in dislocation density during exposure [Figure 5.22] of the K5SC unaged alloy will further exacerbate primary creep. The mechanism responsible for the formation of hard mode dislocations during exposure is not clear. We can speculate on the possible scenarios.

There are several interfacial defects on the \( \alpha_2/\gamma \) interface. Dislocations of the \( 1/2<110 \) type and/or individual \( 1/6<112 \) partials presumably accommodate the lattice misfit across the interface [24-26]. Ledges equivalent to Shockley partial dislocations have been observed by several authors [Figure 5.38] [24, 27, 28]. These have been associated with the transformation from \( \alpha_2 \) to \( \gamma \). Several authors have also observed dislocations with Burger's vector that are not contained in the interface plane. [26, 27, 29, 30]. Small misorientations of 2-5° enclosed between the matching \( (111)_\gamma \) and \( (0001)_{\alpha_2} \) planes, have been observed by Zhang et al [29, 30]. It has been suggested that ledges with a \( 1/3[111] \) Frank partial dislocation component accommodate this misorientation (Figure 5.39). On a \( (111) \) interface plane, the interaction between the Shockley partial and the Frank partial could give rise to ordinary hard-mode dislocations (without change in energy) within the gamma matrix as shown:

\[
a/6 [112] + a/3 [111] \rightarrow a/2 [110]
\]
This provides a possible explanation for the increase in dislocation density inside the γ matrix, after aging. It thus appears that long-term aging not only produces precipitates (in the K5SC material), but also eliminates potential sources of hard mode dislocations in both K5 and K5SC alloys.

5.5. Probable tertiary creep mechanisms.

All FL (and PST) alloys in our study display a tertiary creep behavior under compression. The tertiary creep rate is the greatest for N-oriented PST alloy and the unstabilized K5SC alloy. The degradation of the lamellar microstructure and the formation of recrystallized grains appear to be the chief reasons for tertiary creep in these alloys. The liberation of interfacial dislocations associated with the complete dissolution of α₃ is another probable reason for the large tertiary creep rates [Figure 5.27]. The tips of receding α₂ laths appear to act as an ideal sources of matrix dislocations.

In the stabilized (aged) K5 and K5SC alloys, the loss of α₂ during creep is not significant (the volume fraction drops from 8% to 6%). However, α₂ is often observed to be discontinuous (or spheroidized) [Figure 5.40]. When a thin α₂ lath between two γ laths of the same variant completely disappears, the two γ laths coalesce to form a wider lath. Some researchers have observed spheroidized α₂ in the middle of a γ lath further supporting such a coarsening mechanism. This coarsening of the lamellar structure leads to a reduction in back stresses, a gradual increase in the mean free path of the moving dislocations, and the associated increase in creep rates. There is now a large body of evidence now in support of such a coarsening process [1-5, 31]
While the complete loss of $\alpha_2$ leads to coarsening in the K5 alloys, it creates a string of carbide and silicide precipitates at the $\gamma/\gamma$ interfaces or inside $\gamma$ laths in the K5SC aged material (Figure 5.41). The string of aligned precipitates not only provide precipitation hardening, but also retains the "lamellar" nature of the microstructure and makes the microstructure stable. However, ripening of precipitates has been reported by Noda et al [16] and this probably plays a part in the tertiary behavior of the aged K5SC alloys.

The role of twinning on creep deformation of FL alloys is very poorly understood. While some researchers view twinning as the primary creep mechanism, others insist on its necessity for satisfying the von Mises criterion, and yet others consider twinning to be microstructure refining mechanism. In the next section some of these ideas will be briefly discussed.

5.6. Role of Twinning

Unlike many materials, severe twinning is observed during high temperature creep of $\gamma$-TiAL (especially the equiaxed single phase $\gamma$ alloys) and its role in deformation is baffling. A lot of attention has been paid to the phenomenon of twinning in the past decade. The general agreement in the creep community is that the amount of twinning increases with strain and stress [32-34]. Work by Wang et al. [35] seems to indicate that twinning occurs late in the secondary creep stage [36]. Our observations of crept microstructure indicate the formation of twins only at large strains (4% or more) [Figure 5.42]. The total contribution of twinning to macroscopic strain is usually smaller than that due to slip [32, 37-39]
Deformation twinning is typically considered to be a low temperature (and relatively high strain rate) deformation mode where diffusive mass transport does not occur. It is not clear how this mechanism could control a rate and temperature sensitive process such as creep, let alone contribute to substantial strains. It is suggested that twinning plays a more active role in microstructure evolution than in strain contribution (especially in the lamellar alloys). Several investigators [34, 36, 39-42] suggest that the formation of twin interfaces (especially parallel to the lamellar interface) refines the microstructure and thus causes hardening. (Note: The formation of parallel twins that leads to lamellar refinement is observed only in the soft oriented grains). However, the coincident occurrence of minimum creep rate and twinning suggests that twinning is probably a work softening process rather than a work hardening process in fully lamellar alloys. It is possible to envision twinning as a softening mechanism in more than one way:

1. Twinning reorients the twinned section of the γ-phase more favorably for slip than the parent. Particularly in the case of lamellar microstructure, there is a possibility that the twinned lamellar segment is the same variant as an adjoining lamella, thus leading to coalescence and coarsening [Figure 5.43]. Translamellar twin boundaries are high-energy boundaries and thus are mobile. Twin boundary migration has been previously suggested [43] and this too could lead to the coarsening of the lamellar microstructure.

2. The important factor that controls the formation of twins is the internal stress state. The nucleation of a twin requires stress concentration and so twinning can be considered to be a stress relief or an accommodating mechanism.
and compatibility stresses set up at interface boundaries may be relieved by localized deformation via twinning. (It has been shown that in two-phase lamellar alloys with all \( \gamma \) variants, the von-Mises criterion can be satisfied with just ordinary dislocation slip and true twinning)

3. TEM and HREM work by Appel [31] has revealed that twin matrix interfaces contain dislocation networks which act as dislocation sources. Increased twinning could result in an increase in the dislocation density.

5.7. Conclusions

The following general conclusions can be drawn from our study of the stability of lamellar microstructure under creep conditions

a) Stabilizing the microstructure prior to creep is extremely useful in depressing creep rates during primary, secondary and tertiary creep regimes. Retention of metastable \( \alpha_2 \) during creep can lead to dislocation generation, the loss of lamellar microstructure and the formation of recrystallized \( \gamma \) grains. A detailed study of the effect of orientation on the tertiary creep behavior and microstructures will shed more light on the dynamic recrystallization process and its role in tertiary creep.

b) The addition of C and Si has a substantial effect in reducing the strain rates at all stages of creep by precipitation hardening. These precipitates help in checking the primary creep rates by making soft mode deformation difficult. Through the entire course of creep, the operation of dislocation sources at the lamellar interfaces appears to be choked by the presence of interfacial precipitates. Large primary creep strains have been the greatest concern in the use of fully lamellar alloys for high temperature
applications. Designed microstructures with interfaces decorated with precipitates offer the greatest promise for high temperature applications.

c) While the continued loss of $\alpha_2$ and the concurrent coarsening of the lamellar microstructure are the main source of weakening in the tertiary creep regime, the effect of twinning on microstructure coarsening, and in stress relief cannot be ruled out. A systematic study into the role of twinning is warranted.
LIST OF REFERENCES


Figure 5.1. TEM micrograph of FL K5 alloy in the as-solutionised condition.

Figure 5.2. TEM micrograph of FL K5 alloy after a 24 hr aging treatment at 900°C. Thinning and loss of $\alpha_2$ is evident.

Figure 5.3. Distribution of $\alpha_2$ lath spacings before and after aging.
Figure 5.4. Distribution of $\gamma$ lath spacings before and after aging. Increase in the number of ultrafine $\gamma$ laths observed.

Figure 5.5. a) Formation of ultrafine gamma laths that are arranged in packets, and b) Packet formation appears to take place by the growth of secondary gamma nucleii, as shown. The growth of one such gamma lath terminated by the B2 precipitate.
Figure 5.6. Precipitation of B2 phase associated with dissolving $\alpha_2$ laths

Figure 5.7. TEM micrograph of FL K5SC alloy in the as-solutionised condition.
Figure 5.8. Distribution of $\alpha_2$ & $\gamma$ lath spacings before and after aging. Increase in the number of ultrafine $\gamma$ laths observed in this case as well.
Figure 5.9. Formation of H-type carbide and $\zeta$-type silicide precipitates along dissolving $\alpha_2$ laths [10].

Figure 5.10. The creep behavior of the K5 and the K5SC alloy in the aged condition at the same stress level.

Figure 5.11. The strain rate-strain plot for the above creep tests.
Precipitates
Lamellar Interface
Dislocation interaction with a string of precipitates in the middle of a gamma lath (formed by the complete dissolution of α2 flanked by gamma laths of the same variant type).

Figure 5.12. Crept microstructures of the K5SC alloy indicate extensive interaction between the precipitates and the dislocations at the interface.

Figure 5.13. The different ways in which precipitation strengthening is manifested.
Figure 5.14. Strings of precipitates found in the middle of $\gamma$ laths. These precipitates help in obstructing the motion of dislocations within the $\gamma$ laths and this is seen in the frequent pinning and bowing of dislocations at these precipitates.

Figure 5.15. The creep behavior of K5SC (aged) material is compared with K5SC (unaged) and K5 (aged) at 315 MPa and 760 °C. The K5SC aged alloy is superior to the other two.
Figure 5.16. TEM micrograph of the unaged K5SC alloy

Figure 5.17. TEM micrograph of the K5SC alloy exposed at 815°C for 14.5 hrs. Loss of $\alpha_2$ and formation of packets observed

Figure 5.18. The $\alpha_2$ volume fraction drops from 29% to 10% with time
Figure 5.19. The distribution of $\alpha_2$ thickness indicated as a function of exposure time.

Figure 5.20. Thinning of $\alpha_2$ during exposure.
Figure 5.21. Change in $\gamma$ spacing distribution with exposure time. There is a definite increase in the number of very fine $\gamma$ lamellae.

Figure 5.22. TEM micrographs of the K5SC alloy a) before exposure and b) after exposure at 815°C for 6 hrs. An increase in dislocation density is observed after exposure, even when no stress was applied.
Figure 5.23. The distribution of $\alpha_2$ thickness indicated as a function of exposure time and strain. Samples crept at 150 MPa

Figure 5.24. The distribution of $\gamma$ lath spacing indicated as a function of exposure time and strain. Samples were crept at 150 MPa
Figure 5.25. The distribution of $\alpha_2$ thickness indicated as a function of exposure time and strain. Samples crept at 300 MPa.

Figure 5.26. The distribution of $\gamma$ lath spacing indicated as a function of exposure time and strain. Samples were crept at 300 MPa.
Figure 5.27. $\alpha_2$ dissolution accompanied by the generation of interfacial dislocations. Sample exposed at 815C, 315 MPa for 14.5 hrs

Figure 5.28. Formation of ultrafine laths is observed. Sample exposed at 815C, 150 MPa for 30 hrs

Figure 5.29. Change in $\alpha_2$ volume fraction as a function of exposure time
Figure 5.30. Change in $\alpha_2$ volume fraction as a function of strain.

Figure 5.31. $\alpha_2$ volume-fraction indicated as a function of time for the crept and the exposed samples.
Figure 5.32. Average $\alpha_2$ thickness indicated as a function of time for the crept and the exposed samples. The crept samples appear to retain “thicker” $\alpha_2$.

Figure 5.33. Formation of recrystallized gamma grains in the vicinity of ultrafine packet structure. Recrystallized grains as large as 10 microns have been observed.
Figure 5.34. The interfacial a) (111) plane of $\gamma$ and b) basal plane of $\alpha_2$ are indicated. The darker atoms are the Ti atoms, while the lighter atoms are Al. It is clear that both materials maintain stoichiometry along in these planes.

Figure 5.35. Lower minimum creep rates but poorer tertiary creep behavior observed for the N-orientation [22]

Figure 5.36. The difference in tertiary creep behavior between the two orientations is even more dramatic. [23]
Figure 5.37. Our results are identical to those obtained by the others. Lower minimum creep rate but poorer tertiary behavior for the N orientation under compression.

Figure 5.38. HREM image of Shockley type “transformation” ledge dislocations. Passage of one of these on every other interface plane (along with diffusion) causes $\alpha_2 \rightarrow \gamma$ transformation [27]
Figure 5.39. HREM image of “misorientation” ledge probably similar to a Frank partial [27].

Figure 5.40. Total volume fraction of $\alpha_2$ lost during creep may not be significant in the stabilized microstructures. However, $\alpha_2$ is often observed to be discontinuous (or spheroidized)
Figure 5.41. Strings of a) B2 precipitates in the K5 alloy and b) carbide/silicide precipitates in the K5SC alloy, retain the “lamellar” nature of the microstructure and prevent it from disintegrating completely.

Figure 5.42. BF image (g=(111)) of K5 aged sample crept at 315MPa, 760°C, 4.7% strain. Trans-lamellar twinning is seen at these large strains. Coarsening of lamellar microstructure (effectively the lamellar spacing is widened) due to twinning and recession of $\alpha_2$. 

Figure 5.42. BF image (g=(111)) of K5 aged sample crept at 315MPa, 760°C, 4.7% strain. Coarsening due to twinning and recession of $\alpha_2$. 

Figure 5.42. BF image (g=(111)) of K5 aged sample crept at 315MPa, 760°C, 4.7% strain. Coarsening due to twinning and recession of $\alpha_2$. 

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Table 5.1. The matrix of exposure and creep tests conducted to probe the effect of temperature and stress on microstructural stability
6.1. Conclusions

The creep behavior of an equiaxed microstructure of Ti-48Al was investigated. Stress exponent values in the range of 5-6 were obtained from monotonic creep tests and stress increment tests at 1041 and 1088 K. TEM analysis indicates that the deformation microstructure is dominated by unit 1/2[110] type dislocations, and that these dislocations are pinned by jogs of varying heights. The jogged-screw model was adopted, where the rate-controlling step is assumed to be the non-conservative dragging of the jogs along the length of the screw dislocations. The parameters and functional dependencies assumed in the model have been verified and validated. The original solution has been reformulated to take into account the finite length of the moving jog. This is a better approximation of the tall jog. The substructural model parameters have been investigated in light of the Finite Length Moving Line (FLML) source approximation. By accounting for the fact that there are three competing mechanisms (jog dragging, dipole dragging, dipole bypass) possible, we have arrived at a modified critical jog height beyond which the jog is not dragged. The critical jog height was found to be strongly stress-dependent. Dynamic simulation using a line tension model has shown that the jog spacing is inversely proportional to the applied stress. This has also been confirmed by TEM measurements of jog spacings over a range of stresses. Taylor's expression for the dependence of dislocation density on the
applied stress, was confirmed by actual dislocation density measurements. Combining all of these parameters and dependencies, derived both from experiment and theory, leads to an excellent prediction of creep rates and stress exponents.

$\frac{1}{2}\langle 110 \rangle$ dislocations also dominate the deformation microstructure in FL samples. However, dislocation activity is inhomogeneous, with extensive activity in thicker lamellae and little or no activity in thinner lamellae. It is proposed that there exists a critical lamellar thickness, which is related to the minimum stress required to cause channeling of dislocations: lamellae thinner than the cutoff thickness experience no effective stress. Stress drop experiments indicate large backflow strains in the case of FL and hard oriented PST material and minimal backflow for the equiaxed alloy, confirming the presence of large back stresses unique to the lamellar microstructure. Additionally, these also suggest that primary creep is controlled by soft-mode deformation while the hard mode deformation determines the steady state creep rate. The observation of jogged hard-mode dislocations in the thicker lamellae suggests that a refinement of the modified jogged-screw velocity law for the FL structures may be developed. A procedure to produce such a refinement of the model has been discussed. To calculate the creep rate, it is required that the strain rate be averaged over the range of lamellar spacings. An isostress averaging scheme has been adopted in view of the fact that the FL materials' creep behavior most resembles that of the N-oriented PST sample. This isostress model predicts higher stress exponents and lower creep rates for finer lamellar distributions, in accord with observations. A probable low stress mechanism involving the intrinsic sliding of lamellar interfaces has been proposed. The predicted creep rates are in reasonable agreement to experiment.
Microstructural changes during the aging process in K5 and K5SC alloys have also been studied. Aging causes the dissolution of metastable $\alpha_2$. The final $\alpha_2$ volume fraction in the aged sample was found to be around 8 vol% for both K5 as well as K5SC samples suggesting this to be the "equilibrium" volume fraction. There is minimal effect of Si and C in stabilizing $\alpha_2$. Extensive precipitation of the silicide and carbide particles is seen during aging of the K5SC alloy. These particles were seen to interact with the dislocations along the interface and within the lath and caused pinning. This strengthening is clearly seen in the better creep response of the K5SC (aged) samples when compared to the K5 (aged) and the K5SC (unaged) alloys with no precipitates. The K5SC (unaged) alloy has a creep response poorer than both K5 (aged) and K5SC (aged), which leads to the conclusion that the solute strengthening effects of C and Si are minimal. Rather, the presence of excess, metastable $\alpha_2$ in the microstructure during creep may be detrimental, despite the fact that $\alpha_2$ is a stronger phase. The volume increase during the course of transformation appears to be the cause for dynamic recrystallization. It is concluded that stabilizing the microstructure prior to creep is extremely useful in depressing creep rates during primary, secondary and tertiary creep regimes. Retention of metastable $\alpha_2$ during creep can lead to dislocation generation, the loss of lamellar microstructure and the formation of recrystallized $\gamma$ grains. Stabilized FL alloys with precipitates along the interface appear to be most attractive for high temperature structural applications.
6.2. Scope for future work

There is scope for further validation of several parameters in the jogged-screw model. There is a need for the determination of actual distributions of jog height as a function of stress and temperature via TEM observations. The current understanding of the transient behavior is weak. Stress drop tests on the equiaxed material suggests that there exists an incubation period after the drop [Figure 6.1] during which the jogged-screw model parameters probably readjust to the new stress level. While an increase in jog height slows down the dislocations, beyond the critical jog height, tall jogs start acting as dislocation sources and will accelerate creep rates. It is thus crucial to map out the evolution of jog height, jog spacing and dislocation density with strain. This will help provide an explanation for the observed "normal" primary creep transient for this glide mobility controlled process. A refined understanding of the transient behavior will help in modifying the dynamic simulation efforts. This will also help us in identifying the key microstructural parameters that need to be characterized and reduce the number of experiments to be performed.

Model parameters from the equiaxed microstructure have been adopted for the FL microstructure. A systematic study of the various model parameters in the FL alloy will be very useful. Though the model for FL microstructure is effective in predicting general trends, the actual creep rates predicted are not accurate. This appears to stem from a) the assumption of isostress averaging: using the jogged-screw model to provide constitutive equations for FEM models of lamellar microstructures will confirm the validity of the isostress assumption, and b) the lack of a good representation of lamellar spacing.
distribution: thorough characterization of lath spacings and a better representation of the lamellar distribution will be useful in addressing this source of error.

The low stress regime in the FL alloys needs to be systematically explored with monotonic tests, instead of stress jump tests. Additional TEM study is necessary in order to determine whether an actual change in the principal deformation mechanisms is responsible for the markedly different stress dependence observed in this regime. Fiducial line studies can be employed to explore the possibility of interface sliding. The impact of twinning on the overall creep behavior needs to be systematically explored. The effect of solutes on the model is also not clear. The addition of Cr, Nb and W not only affects vacancy diffusion, but also provide some solute strengthening. The effect of these alloying additions on the CSF energy has not been explored. If alloying additions increase the CSF energy, then the tendency for cross-slip and for the formation of tall jogs is increased. This may be an efficient way of depressing creep rates.

Though the yield strength anisotropy in PST crystals is well documented, the volume of work devoted to high temperature creep of PST crystals is remarkably small. There is ample potential for gaining fundamental understanding of creep mechanisms in lamellar microstructures by doing a systematic creep study on PST crystals. PST crystals also provide a suitable system for exploring anisotropy of microstructural stability. The possibility of tension-compression asymmetry originating from this anisotropy begs to be explored.

The validity of the jogged-screw has been demonstrated in γ TiAl alloys. However this mechanism is not restricted to this particular system. The prerequisites for the formation of tall jogs are that the screw dislocation be compact such that cross-slip is
relatively easy, and that the screw orientation is strongly favored. These prerequisites are fulfilled in several systems. The applicability of the model has been demonstrated for creep in \(\alpha\)-Ti and \(\alpha + \beta\) Ti alloys [1, 2]. a-type jogged-screw dislocations have been observed on both the basal and prism planes. In a recent work, Xu et al. [3], have demonstrated jogged-screw configurations for a\(<100>\) type dislocations in NiAl with B2 structure. Both a-type dislocations in Ti alloys and \(<100>\) dislocations in NiAl, have a compact screw dislocation with penchant for frequent cross-slip. In view of these observations, BCC solid solutions appear to be likely candidates for super-jog formation. The preferential alignment of dislocations along the screw orientation has been attributed to a non-planar three-fold spreading of the \(1/2\langle111\rangle\) dislocation core [4-9]. Cross-slip is frequently observed. A low-temperature microstructure characterized by long jogged screw segments, cusps, debris loops and dipoles is often observed in various bcc metals like Tantalum [8, 10], Niobium [4-6, 11], Molybdenum [9, 12, 13], Iron [4, 14] and Tungsten [15]. It will be interesting to explore creep mechanisms and to evaluate the applicability of the jogged-screw model in these alloy systems.
LIST OF REFERENCES

An incubation period is observed after a stress drop in the equiaxed $\gamma$-TiAl alloy. This behavior has been observed for different stress drops.

Figure 6.1.
APPENDIX A: CALIBRATION CURVES

Figure A.1. Furnace Heating Curve.  
Figure A.2. Thermocouple calibration curve

Figure A.3. LVDT Calibration Curve  
Figure A.4. Load Cell Calibration Curve
APPENDIX B: EXTENDING THE JOGGED-SCREW MODEL TO $\alpha$-Ti

As seen in Figure B.1., the creep microstructure in the near $\alpha$-Ti alloy crept at 538 C and 250 MPa, is similar to that in $\gamma$-TiAl, consisting of cusped screw dislocations. The cusps have been identified as tall jogs. The origin of tall jogs in these alloys is the same as in $\gamma$-TiAl: sluggish and straight screw dislocations with a penchant for cross-slip. Applying techniques similar to those employed for $\gamma$-TiAl we can predict the creep rate using the jogged-screw model.

The dislocation density information for Orowan’s equation has been culled from experimental data for $\alpha/\beta$ Ti alloys. The jog spacing is proportional to the Orowan bowing radius which in turn is related to the shear modulus and the applied stress. Assuming the same jog spacing dependence on applied stress (as in the case of $\gamma$-TiAl) and scaling it according to the shear modulus of $\alpha$-Ti at creep temperature, we can come up with an equation for the jog spacing. The jog height derivation is identical to that employed for $\gamma$-TiAl: by comparing the minimum stress required for jog dragging and dipole bypass. The critical jog height dependence on stress is shown in Figure B.2. The actual tall jog heights measured in $\alpha/\beta$ Ti alloys is comparable to the critical jog height predicted. The predictions of the model for a $\beta$ value of 1 are shown in Figure B.3. The predictions are in close accord with the experimental values thus providing additional support for the model.
Figure B.1. Creep microstructure of Ti-6Al $\alpha$-Ti alloy indicating cusped screw dislocations.

Figure B.2. The critical jog height variation is indicated. Predicted critical jog heights compare favorably with experimentally measured heights.

Figure B.3. The prediction of the jogged-screw model for $\alpha$-Ti is excellent, lending further support to the model.
APPENDIX C: ISOSTRAIN AVERAGING

As can be seen from Figure 4.29, both hard orientations are almost equally creep-resistant compared to the soft orientation. This observation impels us to consider the case when creep in FL alloys is controlled by the A-oriented grains. This represents the other limiting case of isostrain averaging (in reality "isostrain-rate averaging"). The approach is to calculate the stress $\sigma(\lambda, \dot{\varepsilon})$ required to deform a lath of given thickness, at a fixed strain rate. Since the back stress increases for narrower laths, the applied stress has to be much higher in these laths, so that the same strain rate can be maintained. The stress $\sigma(\lambda, \dot{\varepsilon})$ is computed numerically by an iterative method. When the lath thickness becomes very small, the stress $\sigma(\lambda, \dot{\varepsilon})$ required to sustain the creep rate (via jogged-screw motion) is extremely large. It is easier for these thin laths to deform by duffusional creep (Section 4.6) rather than by dislocation creep. So at a critical cutoff denoted by $\lambda_c$, both mechanisms are able to enable the applied strain rate. The iso-strain-rate averaging scheme is indicated by the equation:

$$\sigma_{avg} = \frac{\int_0^{\lambda_c} F(\lambda) \cdot \sigma(\lambda, \dot{\varepsilon})_{\text{diffusional}} \cdot d\lambda + \int_{\lambda_c}^{\lambda_{max}} F(\lambda) \cdot \sigma(\lambda, \dot{\varepsilon})_{\text{dislocation}} \cdot d\lambda}{\int_0^{\lambda_{max}} F(\lambda) \cdot d\lambda}$$
where $\sigma_{\text{avg}}$ is the average stress computed for the imposed strain rate of $\dot{\epsilon}$. The solutions are obtained by numerically solving for the values of $\sigma(\lambda, \dot{\epsilon})$ and $\lambda_c$. The results are shown in Figures C.1. and C.2. for the coarse and fine binary alloys. Also shown are the results from the isostress averaging scheme. It is clear that the strain-rate predicted by the isostrain-rate assumption is lower and in much better agreement with the experimental values for both the fine and coarse lamellar binary alloys, when compared to the isostress approach. The implications of this are not quite clear right currently. It appears that the behavior of the FL material would realistically lies between isostress and isostrain limits.

Figure C.1. The isostrain-rate and isostress predictions are shown for the coarse lamellar binary alloy. The isostrain-rate assumption predicts a better fit than the isostress assumption.
Figure C.2. The isostrain-rate and isostress predictions are shown for the coarse lamellar binary alloy.
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