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UMI®
LOW TEMPERATURE CREEP OF TITANIUM ALLOYS:
MICROSTRUCTURE, DEFORMATION MECHANISMS AND MODELING

DISSERTATION

Submitted in Partial Fulfillment of the Requirements for the Degree

Doctor of Philosophy in the Graduate School

of The Ohio State University

By

Neeraj Srinivas Thirumalai, B.E., M.S.

* * * * * * *

The Ohio State University

2000

Dissertation Committee:

Michael J. Mills, Adviser

Glenn S. Daehn

Hamish L. Fraser

James C. Williams

Approved by

Adviser
Dept. of Materials Science
and Engineering
ABSTRACT

Primary creep is the dominant mode of deformation during ambient temperature creep in titanium alloys. In this study the mechanisms causing this unusual creep behavior have been investigated both at the phenomenological level and at the microstructural level. Main focus of this work is on a single phase α titanium alloy, Ti-6wt%Al(Ti-6Al). Room temperature creep in a commercially important alloy, Ti-6Al-2Sn-4Zr-2Mo(Ti-6242) was also investigated.

Neutron diffraction was used to characterize the presence of SRO in a Ti-6Al alloy. These results indicated there is significant short-range order in this alloy after a conventional treatment like an air cool. From the position of the diffuse peak due to SRO, it was deduced that the short-ranged ordered state is related to the ordered phase Ti₃Al (α₂) with DO₁₉ structure.

Based on the study of both Ti-6Al and Ti-6242 it is shown that the strain rate sensitive Hollomon law, $\sigma = K\dot{\varepsilon}^{n}$, represents the constant strain rate behavior of titanium alloys reasonably well, while the transient creep behavior can be described by a power law of the form $\varepsilon = At^\omega$. A simple analytical result is derived to relate these two expressions. Using this solution the long time creep response has been predicted reasonably well from
constant strain rate results for the two alloys studied. Relative to other metals it is shown that titanium alloys exhibit exceptionally low values of strain hardening.

Effect of SRO on the room temperature creep of a Ti-6Al alloy was studied. Significant differences in the initial transient was observed among various treatments and the final transient was similar among the different samples. Strengthening due to SRO was evaluated quantitatively using dislocation structures. Such an evaluation revealed that the applied stresses are above the friction stress due to SRO, which is very different from the observations in fcc based alloys. Operative slip systems, as well as dislocation distributions and morphologies, are also presented following creep of a single phase alloy Ti-6Al. Both optical microscope observations of slip line evolution and studies of dislocation structures using transmission electron microscopy have been used to relate the deformation mechanisms to the macroscopic behavior.

Weak-fringing faults were observed in <a> type slip bands. Detailed characterization of the faults revealed that they are caused by a residual displacement of the type l/n<11\overline{2}0>. Magnitude of the displacement was evaluated to be between 1/145 to 1/104<11\overline{2}0>. using CUFOUR. Additionally, it was revealed that the dislocations produce a displacement larger than the lattice translation along the <a> direction.

During the course of this investigation additional room temperature deformation anomalies like tension/compression asymmetry, room temperature recovery phenomenon and negative creep have been discovered. The possible sources of these anomalous behaviors have been analyzed, and future directions to improve our understanding of these effects have been identified.
Dedicated to my Parents
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VITA

December 5, 1970

Born - Madurai, India

1992

B.E. Metallurgical Engineering,
P.S.G. College of Technology, Coimbatore

1993-1994

Teaching Associate, Texas A&M University,
College Station, Texas

1994 – Present

Graduate Research Associate, The Ohio State University

PUBLICATIONS


**FIELDS OF STUDY**

**Major Field:** Materials Science and Engineering

**Undergraduate Degree:** Metallurgical Engineering
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CHAPTER 1

INTRODUCTION

Titanium and its alloys are important structural materials used extensively in the aerospace industry, energy industry, chemical industry and as bio-implants. They are very attractive structural materials because of their very high strength to weight ratio and also because of their excellent corrosion properties. In the last 40 years, it has been firmly established that titanium alloys exhibit extensive creep at room temperature. This behavior is very unusual because it occurs at temperatures below 0.2T_m of the alloy. Further, it has been established that significant creep occurs at stresses well below the macroscopic yield strength of the material (as low as 60% of the yield strength). Such ambient temperature creep can occur for very long times. In many cases few percent strain can be accumulated during ambient temperature creep. It has been reported that low temperature creep lead to premature failure of components. Hence, this is not only a significant problem scientifically, but is also a very relevant engineering problem.

In the literature, it has been established that this ambient temperature creep involves only dislocation processes and diffusion is not important. There have been many studies of this room temperature creep behavior on many different alloys. But the underlying mechanisms controlling the process has not been completely understood. The main
phenomenological reason that has been proposed is the high strain rate sensitivity of titanium alloys. Suggesting, that this is the reason for the occurrence of room temperature creep at stresses well below the yield strength. Two microstructural arguments have been proposed, one is based on the ease of operation of dislocation sources and the other is based on slip length arguments. In the first argument it was proposed that under certain microstructural conditions dislocation sources are easy to operate and therefore, leads to extensive creep. Such dislocation source based models usually predict a logarithmic creep law. This is not observed in titanium alloys. Also there is no clear microstructural rationale for the dislocation source argument. The second argument essentially states that when the slip length (i.e. the distance between barriers for dislocation motion), decreases, the total creep strain is reduced. Based on this the poor creep response of a Widmanstätten microstructures was rationalized. It was stated that since there is a Burgers orientation relationship between the α and β laths within a colony, the colony size is the microstructural slip length for Widmanstätten microstructures. Since colony sizes are relatively large, these microstructures would exhibit poorer creep properties. In contrast, in the basketweave microstructure, colony sizes are small and there are many interfaces with no Burgers orientation relationship. In this case slip length is small and hence, such microstructures exhibit good creep resistance. Recently, it has been shown that even in colony microstructures there is a significant anisotropy between the various <a> type slip systems that can operate. Hence, it is not reasonable to assume the colony size to be the slip length. Further, the smaller slip length in the basketweave microstructure does not increase the yield strength significantly.

Titanium-aluminum alloys exhibit a transition in slip from homogenous slip to planar slip for compositions above about 4wt% Al. Many commercial α, near-α and α/β titanium
alloys contain around 6wt% Al. The deformation behavior is usually planar in the \( \alpha \) phase of commercial titanium alloys. Such planar deformation is thought to be promoted due to the presence of short-range order in alloys containing \( \leq 6\text{wt}\% \) Al. However, the effect of short-range order and planar slip on room temperature creep has not been studied. No direct diffraction evidence for the presence of short-range order has been provided in the literature.

The main focus of this work will be to develop an understanding of room temperature creep both at the phenomenological and at the microstructural level. Short-range order in a Ti-6wt% Al alloy will be characterized using neutron diffraction. Finally, the effect of short-range order on room temperature creep will be presented.
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Titanium (Ti) and its alloys are very important structural materials. They are used extensively as structural components in the aerospace industry, as bio-implants etc. because of their high strength to weight ratio[1, 2]. However, it is now firmly established that these alloys exhibit extensive primary creep at temperatures lower than $0.2T_m$, where $T_m$ is the melting point of the alloy in degrees K[3-18]. It has also been shown that this creep behavior can occur at applied stresses below the 0.2% yield strength(YS) of the alloy[4-8, 11-13, 15, 16]. One of the applications in the industry where a component is loaded very close to their 0.2% yield strength is fasteners. The low temperature creep deformation of titanium alloys could be a serious problem in the titanium fastener industry. In fact, in a recent conference on titanium alloys this was identified to be the problem limiting the maximum load of application for titanium alloy fasteners[19]. Low temperature creep been known to pose problems in component tolerencing in the aerospace and gas turbine industry[20]. But under certain conditions they also cause premature failure of the components[20]. Recently, there has been a renewed interest in understanding this phenomenon in the nuclear fuel waste management community[21]. Titanium alloys are being considered as corrosion resistant enclosures for spent nuclear
fuel. These will be stored underground at a depth of 500-1000m in a vault inside a rock formation. Creep at low temperatures (about 100°C) is considered very important because the containers are expected to survive for 500 years under moderate stresses from underground water and also due to the expansion of clay surrounding the containers[21]. Thus understanding the room temperature creep phenomenon in titanium alloys is not only important scientifically but is also a very relevant industrial problem.

2.2 Background

Pure titanium undergoes an allotropic transformation at ≈883°C from a low temperature hexagonal close packed (hcp) phase, α to a high temperature body centered cubic (bcc) phase, β. Titanium alloys are traditionally classified as α, near α, α-β and β alloys depending on the various phases present. The main focus of this survey will be on the first three kinds of alloys and hence, no discussion will be made on β alloys. In general Al, Sn, Zr, O, C and N are all stabilize the hcp α phase and V, Fe, Cr, Mo, Cu and H all stabilize the BCC β phase[1, 2].

Figure 2.1 shows the various microstructures that can be produced in an α alloy[2]. (1) Equiaxed grains are formed when the alloys are worked and annealed in the α phase field. (2) Quenching from the β phase field produces the hexagonal martensite phase α' in which the original β grains remain clearly delineated. α' is produced by a massive transformation. It contains a high density of dislocations and is composed of colonies of plates or laths separated by low angle boundaries. There is negligible hardening associated with this martensite. (3) Slow cooling from the β phase field causes α to form as widmanstätten plates. In high purity alloys, the structure produced is called serrated α. But if impurities like H or Fe are present, they will produce a 'basket weave'
microstructure. Various microstructures that can be produced in a typical two phase alloy Ti-6wt.% Al-4wt.% V(Ti-6-4) is shown in figure 2.2[1]. As one can see, more complex microstructures can be produced in these alloys and figure 2.3 shows a typical widmanstätten microstructure. Several different interfaces have been identified in the literature, a) Grain Boundary: The arrow in figure 2.2 points to a typical "Grain Boundary". Grain boundary here refers to the prior β grain boundary. There can a number of colonies inside a “β” grain. b) Colony Boundary: The arrow in figure 2.3 points to a typical "Colony Boundary". This refers to the boundary between the various colonies which are produced from the same parent β grain. c) α–β boundary: The interface between the α/β plates in a widmanstätten type microstructure in a two phase alloy.

As mentioned above the low temperature phase, α–Ti has a hcp crystal structure. α–Ti has a c/a ratio of 1.587. This c/a ratio is less than the ideal c/a ratio of 1.633. Figure 2.4 shows the important planes and directions in the hcp structure[22] and table 2.1 gives the packing densities of basal, prism and pyramidal planes for hcp crystals of different c/a ratios[23]. For Ti {1010} prism planes are the closest packed planes and \(<11\overline{2}0>\) is the closed packed direction. Slip in Ti occurs mainly by the {1010} \(<11\overline{2}0>\) prismatic slip system as slip is easiest on this slip system.[23] Pyramidal slip system is observed mainly as a secondary slip system [23, 24] and basal slip is also observed as a secondary slip system.

2.3 Ti-Al phase system

The titanium rich end of the binary Ti-Al phase diagram has been studied by many workers[25-34]. In this work the main focus was on a Ti-6wt% (10.17at%) Al alloy. It is
important to know the phases that will be present in the alloy after various heat treatments.

Crossley [25] was one of the first workers to study the Ti-Al system in detail. He proposed that the two-phase $\alpha/\alpha_2+\alpha$ region extends up to 4wt % Al below 500°C. Blackburn [26] studied the Ti-Al system for aluminum content up to 25at% aluminum. The modified diagram for titanium rich compositions by Blackburn is shown in figure 2.5a. This diagram shows that the $\alpha/\alpha_2+\alpha$ boundary lies at around 12at% Al. Blackburn aged a 10.16at% Al alloy at 400°C for 400h and observed diffuse superlattice reflections in a transmission electron microscope (TEM) after this treatment. He could not image the $\alpha_2$ particles using dark field imaging. It was suggested that these diffuse peaks could be due to short-range order (SRO) of titanium and aluminum atoms or due to $\alpha_2$ particles of size less than 25 Å.

Careful studies of the $\alpha_2$ decomposition in the titanium rich end of the Ti-Al system were conducted by Namboodhiri et al [27] and Shull et al [30]. The phase diagram in the composition range of 6at% Al to 20at% Al determined by both the workers was very similar. Figure 2.5b shows the phase diagram determined by Namboodhiri et al [27], it shows that the Ti6wt% Al (10.17at%) composition lies in the $\alpha/\alpha_2$ two phase field. The $\alpha/\alpha_2$ transus for the Ti10at% Al alloy was determined to be 550°C by Namboodhiri et al [27] and as 540°C by Shull et al [30]. Further, Teytel et al [35] showed using neutron diffraction studies that the $\alpha/\alpha_2$ transus for a Ti-9.3at% Al is at 500°C. Finally, Namboodhiri [29] has shown that there is SRO up to 700°C for a Ti-10at% Al alloy.
Crossley [25] proposed that the $\alpha/\alpha_2$ transus could be shifted by $+25^\circ$C for an increase of 0.1wt% oxygen at constant aluminum content or the transus is shifted to 1at% lower aluminum content at 700$^\circ$C for an increase of 0.1wt% oxygen. Similar shift in the $\alpha/\alpha_2+\alpha$ transus was also observed by Ardakani et al [36]. Lim et al [37] studied the effect of oxygen on the properties of a Ti-8wt% Al alloy and found that $\alpha_2$ precipitation became more homogeneous (from heterogeneous precipitation) as the oxygen content was increased for the same ageing treatment. The $\alpha_2$ volume fraction was also found to increase with oxygen content. Above two factors were interpreted to indicate that the $\alpha/\alpha_2$ transus increases as the oxygen content is increased for the given aluminum content. The mechanical test data from the work of Lim et al indicated that oxygen prefers to partition to the $\alpha_2$ phase and this was thought to support the idea that indicating that oxygen stabilizes $\alpha_2$. From the above discussion it appears that the oxygen content of the alloys used in the study can significantly affect the phase diagram. This would not explain the difference in the $\alpha/\alpha_2$ phase as determined by Namboodhiri et al and Blackburn, because the former used alloys which were low in interstitial content (between 350-700wtppm), where as the oxygen content was much higher in the later study (between 1000-1500wtppm). The origin for the disagreement between the two studies is not clear.

2.4 Deformation behavior of titanium rich Ti-Al alloys

Aluminum is one of the most important alloying elements used in almost all $\alpha$, near $\alpha$ and $\alpha-\beta$ alloys. Figure 2.5 shows the phase diagram of the binary Ti-Al system. Many of the commercial titanium alloys contain between 6wt%(10at%) to 8wt%(13at%) aluminum content. Our main interest is in the Ti rich region of the phase diagram, below 13 at. % Al. It should be noted that there is an ordered Ti$_3$Al phase ($\alpha_2$) present at 25at.%
Al in this system. This phase has a DO_{19} ordered hcp structure. The \( \alpha_2 \) phase is very important because it influences the deformation characteristics of the \( \alpha \) phase Ti-Al alloys when precipitated and the deformation is influenced by short range order (SRO) effects in dilute Ti-Al alloys[38-42].

2.4.1 Influence of Al content on the mechanical properties of binary Ti-Al alloys

One of the earliest works on the mechanical properties of binary Ti-Al alloys was done by Ogden et al in 1953.[43] They performed mechanical tests on Ti-Al alloys upto 50wt.% Al. Results of their tests upto 7wt.% Al will be discussed here. Yield Strength increased with aluminum addition at a moderate rate upto 4wt.% Al, after which further additions of Al had a greater strengthening effect. The greatest increase in strength for 1% of aluminum addition occurred between 4 and 5% aluminum. These results are not in agreement with Rosenberg and Nix,[44] who observed a linear increase in yield strength with aluminum addition. Ogden et al do not provide any explanation as to what causes the aluminum to be less potent as a strengthening agent initially upto 4wt.%.

The flow curve of the Ti-Al alloys were fit by a Hollomon[45] type law by Ogden et al[43].

\[
\sigma = A \varepsilon^n
\]

(2.1)

where \( \sigma \) is the flow stress, \( \varepsilon \) is the strain, \( A \) is material constant and \( n \) is the strain hardening exponent. Table 2.2 shows these constants for the Ti-Al alloys from the work of Ogden et al. The strain hardening exponent (n) was found to decrease with increasing aluminum concentration. In other words, strain hardening which is directly represented in \( n \), decreases with increasing aluminum concentration. No explanation was provided by Ogden et al for this behavior. Sakai and Fine[40] observed work softening in a Ti-5.2at.%
Al alloy single crystal. This was thought to be caused by SRO. It is possible that for the same reason (SRO), the work hardening rates of polycrystalline Ti-Al alloys decrease with increasing Al content.

Blackburn and Williams[38] studied the effect of aluminum addition on the mechanical properties of Ti-Al alloys. They observed $C^2$ dependence of aluminum concentration on the yield strength of binary Ti-Al alloys up to 10at% Al. Beyond that concentration they observed a smaller concentration dependence of the yield strength. These results are very similar to the observations of Ogden et al[43]. Blackburn and Williams[38] based on their deformation studies proposed that the strengthening of aluminum is very strong due to short-range order in alloys containing less than 10at% Al. Conrad[46] studied the data of Blackburn and Williams[38] and that of Ogden et al[43] using Flinn's model for short-range order strengthening[47] and suggested that indeed that significant strengthening of aluminum in titanium comes from SRO strengthening. But his analysis was a simplification of Flinn's model, which itself was developed for FCC alloys. He also assumed that prism slip was main contributor to the yield strength of the alloys. Later Okazaki et al[48] showed that such SRO strengthening cannot be present for alloys containing less than 3at% Al. For such dilute compositions they observed a $C^{1/2}$ or $C^{2/3}$ dependence of the yield strength with aluminum concentration.

Rosenberg and Nix[44] studied in detail the solid solution strengthening of aluminum in Ti-Al alloys. They measured the temperature variation of yield stress ($\tau_{ys}$) in alloys containing up to 15at.% Al. Figure 2.6 show the variation of $\tau_{ys}$ as a function of temperature for all the alloys studied. Addition of aluminum shifted the $\tau_{ys}$ versus T curve to higher stresses compared to CP Ti. There is apparently no shift in the critical
temperature $T_c$, the temperature above which the temperature dependence of yield strength is due to the change shear modulus with temperature, with aluminum addition. In contrast $T_c$ shifted to higher temperatures as the interstitial content was increased in $\alpha$–Ti[49, 50]. (It worth mentioning here that all the alloys tested by Rosenberg and Nix contained approximately 1000 ppm Oeq of interstitials). It was concluded that aluminum contributed mainly to athermal strengthening of titanium. They observed a linear increase in yield strength with aluminum concentration. This is not in agreement with Ogden et al's[43] results, who observed a lower increase in strength with aluminum addition, up to 4wt%Al(7at%Al), and then a much higher increase in strength with further additions of Al. Rosenberg and Nix[44] observed that the dislocations tended to prefer the screw orientation with aluminum addition. No change in dislocation density due to aluminum addition was observed, despite the dramatic change in dislocation character brought about by aluminum addition. This observation is supported by Sakai and Fine[40], who made similar observation in dilute Ti-Al alloys. Hence, Rosenberg and Nix proposed that the athermal strengthening is due to interactions between edge kinks in screw dislocations and the aluminum atoms.

Sakai and Fine[40, 51, 52] did extensive work on single crystal plastic deformation studies of Ti-Al alloys. Single crystals containing up to 5.2 at.% Al were oriented for prismatic slip and studied in the temperature range 77K-600K. Critical resolved shear stress(CRSS) for prismatic slip as a function of aluminum content and temperature is shown in figure 2.7. Alloy softening was observed for aluminum additions up to 0.87at.% Al. Alloys with higher interstitial content showed larger alloy softening (this result is not shown here). This softening was attributed to scavenging of impurities by Al. Alloy softening was not observed by Rosenberg and Nix[44] in their study on Ti-Al alloys, but
the minimum Al content in their study was 3at.% Al. Beyond the minimum in CRSS, Sakai and Fine[40] observed a linear dependence of CRSS with concentration of Al. This is in agreement with results of Rosenberg and Nix. Both the temperature and solute dependence of CRSS is plotted in on a temperature axis figure 2.8 from Sakai and Fine[40]. It is evident that CRSS shows a strong temperature dependence. Small additions of aluminum decreased the temperature dependence of CRSS and it remained constant with further additions of aluminum. Addition of 5.2% Al increased the temperature dependence again. This was thought to be in agreement with the argument that aluminum getters the impurities. Sakai and Fine attributed the temperature dependence of the 5.2at.% Al alloy entirely to aluminum. But this is not in agreement with the conclusions of Evans[53] and Rosenberg and Nix[44] who concluded that Al contributed mainly to athermal strengthening.

Stress-strain plots of the various alloys deformed at room temperature by Sakai and Fine is shown figure 2.9. One important result to note is that the 5.2at.% Al alloy showed work softening. Sakai and Fine attributed this behavior to the destruction of SRO during plastic deformation. This agrees with Ogden et al[43], who observed that increase in aluminum content decreased the strain hardening rate. In addition Sakai and Fine[40] found that the higher interstitial alloys had a lower work hardening rate compared to lower interstitial alloys, although work softening was not observed in those alloys.

Sakai and Fine [52] conducted shear tests to determine the CRSS for basal slip as a function of aluminum content. They observed that the ratio of CRSS for basal versus prism slip decreased as the aluminum content was increased as well as when temperature of deformation was increased. This is shown in table 2.3. Taken from their work. Similar
results were observed by Paten et al[54], who observed an even greater decrease in the ratio of CRSS for basal versus prism slip for even greater aluminum contents. This is shown in figure 2.10. It is not clear why there is a difference between the results of Paten et al and Sakai and Fine in the magnitude of the ratio for similar compositions. It could be due to differences in the interstitial content of the alloys used in the two studies. The interstitial content in the work of Paten et al is not available, but comparing the measured CRSS for compositions similar to that used by Sakai and Fine, it appears that the interstitial content was lower in this study compared to that of Sakai and Fine.

2.4.2 Dislocation configurations in Ti-Al alloys

Sakai and Fine[40] in their higher purity alloys observed that screw dislocations or dislocations near screw orientation predominated the microstructure on samples deformed lightly. The greater density of screw dislocations compared to edge dislocations implies that the velocity of edge dislocations is faster than that of screw dislocations. This is because the Peierls stress for screw dislocations is thought to be larger than that for edge dislocations due to the corrugated nature of the prism plane[55]. In addition the small jogs and dislocation dipoles which are present probably contribute to slowing down of screw dislocations. Metzbower[56] when measuring the stacking fault probability of Ti-Al alloys, concluded that the stacking fault energy (SFE) of close packed (0001) planes is lowered by the addition of Al. This may explain why there is an increase in screw dislocation density with increasing aluminum content, because the screw dislocations would tend to split more on the basal plane. But observation of screw dislocations split on the basal plane have not been made in the TEM so far. There have been numerous atomistic studies on the dislocation core structure of screw dislocations in α-titanium. It was shown that the core structure of screw dislocations is spread on both
prism and basal planes. Hence, there is a larger Peierls' stress on the screw dislocations making them the slowest moving line orientation. This is discussed in section 2.4.4.

Blackburn and Williams[38] did studies on the dislocation structures of both CP Ti and Ti-Al alloys containing up to 25at.% Al. Cell structure was observed to form in the CP Ti (1800 ppm Oeq) sample after 4% deformation. Addition of 5at.% Al (1200 ppm Oeq) lead to a more homogeneous distribution of dislocations with neither well defined cell structure nor planar group of dislocations. Planar group of dislocations predominantly of screw orientation was observed in alloys containing >10at.% Al (see figure 2.11). In the higher aluminum alloys dislocation tangles were observed only at higher strains. The strain required to produce such tangling was observed to increase with increasing Al content. Higher Al contents tended to produce narrower slip bands. The tendency to prefer screw orientation in a dislocation increased with Al content. This is similar to the results of Rosenberg and Nix[44] and Sakai and Fine[40] Occasional cross slip was observed in the lower Al contents (<10at.% Al), but this process seemed to be unfavorable as the Al content increased. Blackburn and Williams argue that the lack of cross slip is not due to lowering of stacking energy with aluminum addition but it is due to SRO effects. Preference for planar slip with increasing aluminum content was rationalized in the following way. In the Ti-Al system an ordered phase Ti₅Al with DO₁₉ structure is present at 25at.% Al[26, 27]. Short range order titanium and aluminum atoms was thought to occur at lower concentrations of Al[26, 27] and presence of SRO was thought to promote planar slip in the lower aluminum content alloys. Such transitions from homogenous to planar slip was observed by Lim et al[37], Paton et al[42] and Williams and Lüjtering[57] around 4wt%.
2.4.3 Effect of ageing on α phase deformation in α/β alloys

Welsch et al [58] studied the α phase deformation behavior after solution treatment and subsequent ageing at lower temperatures. The solution treatment was an anneal at 810°C for 1 h and a water quench. Samples were also aged at 350°C and 550°C for 67h each. They performed constrain strain rate tests in tension after these various treatments. The solution treated samples showed homogeneous deformation and slip was observed in both prism and pyramidal planes. The 550°C aged sample exhibited planar slip, they observed long screw dislocation pile-ups in both prism and pyramidal planes. Since they observed α2 reflections in the TEM they attributed the planar slip due to cutting of α2 particles. In the 350°C aged sample the deformation was found to be planar. The planarity was found to be more severe than the 550°C aged sample. Pyramidal slip was found to be predominant after this heat treatment. They also observed pairing of dislocations. It was not stated whether the pairing was observed at the head of the pile-ups or not. From their figure 9 it appears that the pairing is not at the head of the pile-ups and could be dislocations dipoles. Since they did not observe any α2 reflections in the diffraction patterns in the TEM combined with the fact that diffusivities are very low for both Ti and Al atoms, they argued that oxygen ordering occurs in this condition because of the higher mobility for oxygen atoms and the relatively high oxygen content (0.22wt%) of the alloy. Diffraction information indicating the presence of oxygen ordering was not provided. Oxygen ordering was proposed based on the predominance of pyramidal slip, planar slip, observation of dislocation pairing and the low diffusivities of Ti and Al atoms.

Welsch and Bunk[59] studied the deformation modes in the alpha phase of the Ti-6-4 as function of oxygen content and heat treatment. The samples were beta annealed at
1050°C and then slow cooled to 800°C and oil quenched to room temperature (STQ).

Some samples were then aged at 550°C for 1, 10, 115 h, then air cooled to room temperature. Samples were also aged at 350°C for 1 h and 115 h, then air cooled to room temperature. In the STQ condition they observed homogeneous slip in the low oxygen alloy (O₂ content = 0.07 wt% ) and observed mixture of homogeneous and planar slip in the higher oxygen alloy (O₂ content = 0.19 wt %). In both the alloys the preferred slip plane was found to be prism planes. After the 350°C 115 h age, the low oxygen alloy exhibited homogenous slip on prism planes and the higher oxygen alloy exhibited planar slip predominantly on pyramidal planes. Based on these results oxygen ordering was proposed to cause slip planarity and the attendant change in mechanical properties. Their argument is based on Churchman’s model [24] to explain the difference in CRSS for slip between basal, prism and pyramidal planes in α-Ti as function of oxygen content. Churchman [24] had observed that in α titanium single crystals the difference in CRSS for slip in prism, basal and pyramidal planes decreased as the oxygen content was increased. He found that the CRSS for prism and basal slip increased much more than that for pyramidal slip as the oxygen content was increased. A model was proposed based on the occupancy of the octahedral sites by oxygen atoms. In this model the oxygen atoms occupying the octahedral sites interact more with the dislocations on the basal and prism planes than with dislocations on one of the equivalent pyramidal planes. Even if one assumes the model to be correct in explaining the relative changes in CRSS between the various type slip systems with oxygen content, it cannot be argued that pyramidal slip will be favored over prism or basal slip. The selection of the particular slip system will depend on the Schmid for that system and Churchman’s model only predicts that if both prism and pyramidal slip systems have the same Schmid factor then both can be activated in the higher oxygen alloy, whereas only prism slip will be activated in the
lower oxygen alloy because of lower CRSS for prism slip in the second case. In the 550°C aged samples, low oxygen alloy exhibited weakly planar slip and the high oxygen alloy exhibited planar slip. Prism planes were observed to be the preferred slip planes. The planarity of slip in the higher oxygen alloy was thought to occur due to the cutting of \( \alpha_2 \) particles.

Effect of oxygen and heat treatment on the deformation of single phase alpha alloy Ti-6Al-2V was studied by Liu and Welsch[60]. They studied alloys with oxygen contents of 0.07, 0.2 and 0.65wt%. The samples were solution treated at 40°C below the \( \alpha/\beta \) transus between 840°C and 940°C for the lowest and highest oxygen alloys and water quenched to RT. Samples were subsequently aged at 350°C and 550°C. They observed weak superlattice reflections indicating the presence of \( \alpha_2 \) in both the 0.07wt% O\(_2\) and the 0.65wt% O\(_2\) alloy after a 550°C-50h age. In the 0.65wt% oxygen alloy they observed dislocation pairs (it was not stated whether it was observed at the head of the pile-up or not) even in the as STQ condition. It was also stated that such pairing was not observed after the 550°C ageing when Ti\(_3\)Al reflections were observed in the electron diffraction pattern. This was thought to suggest the presence of oxygen ordering in the high oxygen alloy. But clear pairing of lead dislocations in a pile-up was observed in the presence of \( \alpha_2 \) particles by Blackburn and Williams[38]. Hence, the above argument that dislocation pairing indicated the presence of oxygen order is not complete. Further, it was proposed by Blackburn and Williams[38] that SRO of Ti-Al atoms exist for a Ti-6wt%Al (10at% Al) alloy based on the presence of planar slip in these alloys. This composition is similar to the alloy used by Liu and Welsch[60]. Based on SRO in FCC alloys it was suggested by Cohen and Fine[61] that in the presence of short-range order the lead dislocations will be paired. From the above it is reasonable to suggest that in fact presence of SRO
promoted planar slip in the alloy aged at 350°C, in which there will not be any superlattice reflections present and pairing of lead dislocations will occur.

Oxygen order was proposed in alloys containing significant amounts of oxygen and were aged at 350°C[58-60]. It is not clear whether presence of SRO will promote pyramidal slip. Naka and co-workers[62, 63] have proposed that the core structure of screw dislocations are dissociated on the prism plane and symmetrically on two pyramidal planes in high interstitial α-Ti alloys. Such a core structure was proposed based on the preference for screw line orientation in the dislocations and evidence for frequent cross-slip on to pyramidal planes. This type of core structure was not observed in atomistic studies, where the screw dislocation core was observed to be spread in both prism and basal planes[64-66]. Farenc et al[67] deformed the same alloy used by Naka, in-situ in a TEM and they also observed frequent cross-slip onto the pyramidal planes. So it is possible that the preference for pyramidal slip is due to the presence oxygen and planar slip is due to SRO of Ti-Al atoms in these alloys. But in the work of Williams et al [50] on α Ti-O alloys, they observed a slip transition to planar glide at an oxygen level of 1750 wt ppm in samples deformed at 77K and above. They noted that the preferred slip plane in the planar slip regime is both prism and pyramidal planes and not exclusively pyramidal slip. Planar slip was thought to be promoted by SRO of Ti-O atoms.

From the above discussion it is apparent that the deformation in the α phase of a two phase alloy is very similar to binary Ti-Al alloys with 6 to 8wt% Al. Also the combined effects of oxygen and aluminum on the deformation behavior is not very clear.
2.4.4 Non-schmid effects and \(<a>\) dislocation core structures

Sakai and Fine [51] were the first to report failure of Schmid’s law for prismatic slip in dilute Ti-Al alloys. They observed orientation dependence of CRSS for prismatic slip. Large CRSS values were obtained when the compression axis was less than 55° from the c axis, but the CRSS was found to be within experimental errors when the compression axis was beyond 65° from the c axis. A Ti-0.87 at% Al alloy obeyed Schmid’s law at 500K. There was large temperature dependence of yield strength for the orientation (schmid factor 0.22 for prism slip) with the largest failure of Schmid’s law for aluminum contents ranging from zero to 3.15 at% compared to the orientation with a schmid factor of 0.5 for prism slip. For a 5.2 at% Al content alloy, the compression axis oriented with schmid factor 0.22 for prism slip and with schmid factor 0.5 for basal slip, slip predominantly occurred on basal planes. This was in complete contrast to other composition discussed before. The slip lines were observed to be wavy and extensive cross slip between basal and prism planes was observed. This result was interpreted as that the ratio of CRSS for basal to prism planes decreased with increasing aluminum content (see table 2.3). Sakai and Fine attributed this failure of Schmid’s law to asymmetric core splitting.

Naka et al [63] performed studies on alpha titanium with different amounts of impurities. They also reported an orientation dependence of CRSS for prism slip. This was thought to be an indication of non-planar core structure of \(<a>\)-type dislocations. It was observed that the dislocations were predominantly in screw orientation and had a propensity to cross-slip onto first order pyramidal planes. From these observations, Naka et al proposed that the core structure of \(<a>\)-type screw dislocations are dissociated in three planes namely prism and the two first order pyramidal planes (see figure 2.12). This was
supported by evidence that the orientation for which the largest violation of Schmid's law occurred was also the orientation in which the Schmid factor for one of the conjugate pyramidal planes was close to zero.

Several atomistic studies using many different inter-atomic potentials have been done in α-titanium to study the core structure of <a> type screw dislocations[64-66, 68-72]. The most recent of these studies is by Girshick et al[66]. It was observed that the core of the screw dislocation was spread on both prism and basal planes (figure 2.13). Using a bond order potential which accounts for covalency of inter-atomic bonds, Girshick et al showed that energetically spreading on the prism plane is favored. Similar results were reported by Legrand[64] and by Vitek and Igarshi[65]. It should be pointed out that the core structure proposed based on atomistic studies is very different from the one proposed by Naka et al[63]. In the former the screw dislocation core structure is spread only on two planes, prism and basal. Where as in the latter case the core is spread on three planes, two pyramidal planes and on the prism plane. De Crecy et al[73] studied the core structure of an <a> type edge dislocation using High resolution electron microscopy (HREM). They observed that the core structure is planar and is spread only on the prism plane. The above discussion shows that the edge/mixed dislocations will be more mobile in titanium alloys than the screw dislocations, because the latter experiences a large Peierls' stress due to the non-planar core structure.

2.5 Effect of SRO on deformation behavior

Blackburn and Williams[38] observed dislocation pairs at the head of pile-up structures in planar arrays in alloys containing greater than 13.5 at.% Al. Hence, they concluded that it was SRO that was causing the slip transition from diffuse to planar slip in more dilute
Ti-Al alloys. This kind of slip transition towards planar slip due SRO have been in FCC based alloys as well[74, 75], which is discussed below.

2.5.1 Strengthening due to SRO

It was first proposed by Fisher[76] that SRO can provide significant strengthening in solid solutions. He suggested that one can relate the amount of strengthening to the energy of the disordered interface that is produced when SRO is destroyed due to passage of a dislocation on the slip plane, i.e. \( \tau_{\text{SRO}} = \gamma_{\text{SRO}} \). Since then, there have been several theoretical studies attempting to relate the quantity \( \gamma_{\text{SRO}} \) (called diffuse anti-phase boundary (APB) energy) to SRO parameters. Most of these studies have been on FCC alloys[47, 61, 77-83] and some on BCC alloys[84, 85].

The basic principle of these theoretical studies has been to estimate the number of unfavorable bonds that have been produced due to the slip process, and relate this to \( \gamma_{\text{SRO}} \). In all these studies pair interaction energies have been used and bond counting have been performed ranging from only nearest neighbor violations to farther distances to estimate \( \gamma_{\text{SRO}} \). Cohen and Fine[61] using pair interaction energies and considering only nearest neighbor violations showed that the \( \gamma_{\text{SRO}} \) created by the first dislocation is only partially healed by the second dislocation and subsequent dislocations do not restore the crystal to original equilibrium SRO state. From this Cohen and Fine suggested that pairing of lead dislocations will occur when there is SRO in an alloy. This could be used as an indication for the presence of SRO. The most recent of the studies to estimate \( \gamma_{\text{SRO}} \) is by Schwander et al[83]. They developed a scheme by which any number of SRO parameters and neighbor distances can be used to estimate the quantity \( \gamma_{\text{SRO}} \). Again pair interaction
energies were used and these quantities were estimated from the measured SRO parameters by the Inverse Monte-Carlo method[86]. The change in $\gamma_{SRO}$ with the number of dislocations shearing the slip plane was calculated by Schwander et al for a Ni-11.2at%Mo alloy. They observed that the first dislocation creates a fault and there are some small oscillations of this fault energy for the next four dislocations. After the fifth dislocation, the fault energy is constant and this was identified as $\gamma_{SRO}$. A plot of $\gamma_{SRO}$ as a function of number slip steps is presented in figure 2.14 from the work of Schwander et al. In other words, once the initial SRO state has been altered by the passage of the first few dislocations, subsequent dislocations do not return the crystal to the initial state.

Mohri et al[82] also performed theoretical calculations on a model FCC alloy system, to understand the nature of the friction stress due to creation of the diffuse-APB, $\tau_{SRO}$ on each dislocation as they cut through a slip plane sequentially in an short-range ordered alloy. They noted that the largest friction was experienced by the first dislocation, the second dislocation experienced a small attractive force and the subsequent dislocations experienced a friction stress close to zero. These conclusions are consistent with the work of Cohen and Fine[61]. Based on the above description it follows that the lead dislocation experiences a friction force opposing its motion due to the creation of $\gamma_{SRO}$ whereas the trailing dislocations do not experience any friction stress because they do not alter the SRO structure. The trailing dislocations in the slip plane do experience elastic interaction with the solutes ($\tau_{SS}$). Recently, Patu and Arsenault[87] based on computer modeling of the stress required to move a dislocation through an array of solute atoms on a slip plane, showed that solid solution strengthening in alloys with SRO will be less than that observed in the same alloy with random distribution of solutes. Their studies did not consider the effect of $\gamma_{SRO}$ but only the elastic interaction of solutes with a dislocation line. To recapitulate, strengthening in solid solutions with SRO is mainly due to the
friction stress experienced by the lead dislocations in the creation of $\gamma_{\text{SRO}}$. Glide plane softening occurs after the first few dislocations have cut through the slip plane destroying the SRO. Thus presence of SRO will then promote planar slip because of this glide plane softening effect. Dislocations will tend to move in pile-ups with tens of dislocations in them.

There have been observations that planar slip does not occur in an alloy even though there is significant SRO present in the alloy (for e.g., Ni-11 at%Cr[74]). Stated another way, two alloys could have the same SRO parameters representing the number of unlike nearest neighbors, but one would exhibit planar slip whereas another alloy would exhibit diffuse slip. Schwander et al estimated $\gamma_{\text{SRO}}$ for a similar alloy Ni-11 at%Cr for the corresponding SRO state and found that to be only 3.6 mJ/m$^2$. From this they suggested that the magnitude of $\gamma_{\text{SRO}}$ could determine whether one observes planar slip or not, in solid solutions with SRO. Roelofs et al[88] applied this criterion to various Cu based systems, Cu-Zn, Cu-Al and Cu-Mn to identify the solute content at which transition from wavy to planar slip occurred. They noted that planar slip seems to be promoted when $\gamma_{\text{SRO}}$ is above 2-4 mJ/m$^2$.

Estimation of the friction stress due to SRO ($\tau_{\text{SRO}}$) has also been attempted from the observed dislocation structures in a TEM. Prinz et al[89] attempted to estimate the friction stress due to SRO in Cu-Al alloys from the curved dislocation segments present in the post-deformation structures observed in a TEM. Neuhaüser et al[90] estimated the friction stress in Cu-Ge, Cu-Al and Cu-Zn alloys from the distance of separation between the parallel slip planes forming the multipolar structure. Using this method the friction stress was estimated to be 60%-90% of the observed CRSS at room temperature. Clemen
et al [91] studied the friction stresses due to SRO in Ni-33at%Cr alloy. From TEM micrographs taken during in-situ deformation in a microscope, they noted the dislocation positions in isolated dislocation pile-ups immobilized in the middle of a grain. After making a few simplifying assumptions (see [91] for details), the interaction stresses due to the self stresses of the dislocations in the pile-up was calculated. The stress at the pile-up head was estimated to be 2-3 times the macroscopically measured CRSS. In other words, the friction stress experienced by the lead dislocation was very high, and much higher than the applied stress. Further, they noted that the dislocation distribution deviated from an ideal pile-up at the head, whereas at the tail it was more close to an ideal pile-up. This was interpreted as due to the presence of strong friction at the head of the pile-up and less friction at the tail of the pile-up. From these observations, they concluded that SRO promotes glide plane softening and the formation of dynamic dislocation pile-ups. Also, formation of these pile-ups allows the dislocations to move at lower stress levels because of the stress amplification at the head of the pile-up due to the self stresses of the dislocations present in the pile-up. Estimation of friction stress on isolated pile-ups was performed on post-deformed dislocation pile-ups observed in a TEM foil in Cu-Zn alloys by Olfe and Neuhauser[92], in the γ phase of a Ni-based superalloy and on a Ni-26.5at%Cr alloy (with Mn, Co and W) by Jouiad et al[93]. In both studies they estimated friction stresses at the head of the pile-up to be about twice as high as the macroscopically measured CRSS. Jouiad et al[94] also estimated the friction stresses at the head of a dynamic pile-up under load during in-situ deformation on the Ni-26.5at%Cr alloy (with Mn, Co and W). From these calculations they estimated the friction stresses to be four times higher than the macroscopic CRSS. They showed that estimation of friction stresses from pile-ups in post-deformed microstructures will be an underestimate of the actual friction stresses.
Plessing et al[95] studied the strengthening due to SRO in Cu-Al and Cu-Mn alloys. From the measured SRO parameters they calculated $\gamma_{SRO}$ using the method of Schwander et al[83]. They also estimated $\gamma_{SRO}$ from friction stresses measured from dislocation pile-ups in post-deformed microstructures. Reasonable agreement between the two estimates of $\gamma_{SRO}$ was obtained by them. Jouiad et al[93] observed pairing of only the first two dislocations in their studies of a Ni-26.5at%Cr alloy (with Mn, Co and W) and from the estimation of friction stresses in pile-ups, they calculated a $\gamma_{SRO}$ value for the first and second dislocation. These values were in reasonable agreement with those calculated by Schwander et al[83] for a binary Ni-21.2at%Cr alloy. Although in their subsequent work, Jouiad et al[94] measure $\tau_{SRO}$ to be twice as large as the estimate from static pile-ups in the same alloy. This would imply that $\gamma_{SRO}$ will also be twice as large. On the whole, both theoretical estimates of $\gamma_{SRO}$ and those estimated from pile-up configurations seem to be in reasonable agreement.

2.5.2 Dynamics of dislocation pile-ups, formation and motion

In the previous sections, it was shown that deformation in Ti-Al alloys (for Al content >4wt%) and other SRO forming alloys proceeds by the cooperative motion of many dislocations in the form of pile-ups with tens of dislocation in them. It is important to understand the dynamics of formation of a pile-up and the motion of a pile-up to understand the deformation behavior in such alloys. A number of theoretical studies have been done on dislocation pile-up dynamics, which will be reviewed in this section.
There have been many studies where the dynamics of "free expansion" of a pile-up under an applied stress or under no stress (due to the mutual repulsion of like dislocations) have been studied. Rosenfield and Hahn[96] were the first to perform such calculations. In their work they considered a pile-up of screw dislocations generated from a source and moving away from the source under the action of the applied stress. They assumed a semi-logarithmic relationship for the stress dependence of the velocity of an individual dislocation. The effective stress on the dislocations were due to the applied stress and the inter-dislocation stresses, no friction on the motion of dislocations was considered. The calculations were carried out under a constant applied stress. Velocity of the lead dislocation in the pile-up was found to be as high as 200 times the velocity of an individual dislocation under the same conditions. Effective stress on most of the dislocations in the pile-up was calculated to be greater than the applied stress(=1.5τ_{applied}). The dislocations were found to be piled-up against the source in an "inverse pile-up" configuration. This was rationalized based on the fact that back stresses on the trailing dislocations due to the dislocations ahead of them will work against the applied stress, whereas the self stresses due to the trailing dislocations will amplify the applied stresses on the leading dislocations. Velocity of propagation of the array was found to be much larger than the velocity of an individual dislocation. Assuming that the pre-exponential factor was same for both screw and edge dislocations, they showed that the source producing edge dislocations will operate more rapidly than a source producing screw dislocations. Ratio of the two rates of operation was suggested to increase with increasing stress and increasing stress sensitivity.

Head[97, Head, 1972 #160] considered "free expansion" of a pile-up of fixed number (n) dislocations under many different conditions. A power law was assumed for the stress
dependence of the velocity of an individual dislocation \( v = M \sigma^m \). He showed that similarity solutions exist for the non-linear equations of motion, for both a discrete dislocation distribution and for a continuum approximation of dislocation distribution. In the case of no applied stress, for the expansion of 11 dislocations which started out as a conventional pile-up, the central dislocation of the distribution did not move for all \( m \) values, due to the symmetry of the arrangement. For the limiting case of \( m = 0 \) (no stress dependence), the lead two dislocations were found to be closer to each other and the pile-up stayed as a classical pile-up, but increasing length with time. Whereas for the limiting case of \( m = \infty \), the dislocations were piled up against the central dislocation. He also showed that \( m \) does not have to be very large or very small before the dislocation arrangement resembles the two limiting cases. Symmetry of these arrangements were thought to occur due to the inverse dependence of interaction stresses with the distance of separation between the dislocations. It was shown that these similarity solutions existed only for special conditions. Head[98] and Head and Wood[99] showed that general solutions for this problem is available only in special cases and in other cases one might have to solve the positions of the dislocations numerically.

Yokobori et al[100], considered the case of an edge pile-up emitted by a source and their motion away from the source under constant rate of applied stress. The source activation stress and the friction stress on individual dislocations was assumed to be zero. They assumed a power law relation for the stress dependence of the velocity of an individual dislocation \( v = M \sigma^m \). For these calculations they assumed a \( m \) value of 3. Again, the pile-up was found to be an “inverse pile-up”. It was shown that in this case the effective stress on the dislocations was nearly equal to the applied stress and the velocity of the lead dislocation is approximately 1.65 times the velocity of an isolated dislocation. This result
is very different from that obtained by Rosenfield and Hahn[96] for the constant stress case with a semi-logarithmic stress dependence of the velocity. Yokobori, Jr. et al[101] extended these calculations to other m values ranging from 1 to 6.4. For all the cases, they found that the effective stress on the dislocations in the pile-up was nearly constant and equal to the applied stress. Again the velocity of the lead dislocation was found to be less than twice the velocity of an individual dislocation under the same conditions. Yokobori, Jr. et al[102] further extended these calculations where the source produced an equal number of positive and negative dislocations. Again, a constant stress rate was applied and a power law was assumed for the stress dependence of dislocation velocity. The results were observed to be similar both qualitatively and quantitatively to their two previous calculations.

Arsenault and Kuo[103] considered the motion of a group of dislocations emitted from a source under a constant applied stress. They assumed a power law stress dependence on dislocation velocity \(v=Ma^n\) and no friction stresses on individual dislocations were considered. Calculations were performed assuming no source activation stress and a source activation stress equal to the applied stress, also for m values in the range of 0.5 to 35. For both cases of source activation stresses they found the dislocation arrangement to be piled up against the source. The lead dislocation velocity did not exceed 2.5 times the velocity of an individual dislocation in any of the cases calculated. This result is in disagreement with the results of Rosenfield and Hahn[96]. However, the authors showed that if reasonable formulation of source activation was included in Rosenfield and Hahn's formalism, one would get lead dislocation velocities that are 1.5 to 3 times that of an individual dislocation.
Patu and Shih[104] considered dislocation group motion under the action of a constant forward stress and reverse stress. Results of only the case of forward stress will be discussed here. They assumed a power law relation for the stress dependence of dislocation velocity \( v = M \sigma^m \); \( m \) values ranged from 1.02 to 1.49. Friction stresses on the dislocations were not considered. Annihilation of a dislocation at the source if the effective stress of the dislocation was negative and it moved to the location of the source was considered. Again, the dislocations were observed to be piled up against the source. The average velocity of the dislocation group was found to be the same as the velocity of an individual dislocation under the same applied stress. The lead dislocation velocity was observed to increase rapidly with addition of dislocations in the group up to the sixth dislocation in the group, after which the velocity was found almost constant at a value of approximately 1.5 times the velocity of an individual dislocation. It was also shown that the emission rate of the source decreases with the number of dislocations in the pile-up. They also considered the motion of a dislocation group with \( n \) number of dislocations under no applied stress. Contrary to the work of Head[97] they allowed the number dislocations in the group to change. The motion of the group of dislocations under their mutual repulsion was observed to be very complex. At the end away from the source the dislocations expanded. But, at the end near the source the dislocations moved towards the source and annihilated at the source. Movement of dislocations in the middle of the band was found to be very complex.

Arsenault and Cadman[105] performed calculations on thermally-activated motion of a group of dislocations. The dislocations were assumed to overcome short-range barriers to their motion by thermal activation. Three kinds of short-range barriers were chosen for the simulations, one was called weak short-range barrier, the second was based on
experiments on a Fe-Si alloy and the third barrier was based on experiments on Nb. The calculations were carried out under a constant applied stress for different number of dislocations. For the case of Fe-Si alloy with eleven dislocations in the pile-up, they observed that the velocity of lead dislocation was 1.65 times faster than single dislocation traveling through the same short-range barriers. It was shown that the dislocations do not stay in an inverted pile-up configuration, as it was observed in other studies of free expansion of a pile-up. The reason for this was the average velocity of the lead dislocations slowed and approached the velocity of a single dislocation. This is in contradiction with the statement that the steady state velocity of the lead dislocation was higher than the velocity of a single dislocation. The lead dislocation velocity for the Nb barrier was found to be less 2 times the velocity of an individual dislocations and for the weak short-range it was 2.63. For the case of the weak short-range barrier the dislocation pile-up resembled an inverted pile-up. Arsenault and Cadman also conducted simulations of motion of two-dimensional arrays. In this case the dislocations were in an inverted pile-up configuration. The velocity of the lead dislocation was calculated to be twice the velocity of a single dislocation. Finally, it was noted that the average velocity of the dislocation group was the same as the velocity of a single dislocation.

Rosenfield and Hahn[106] also calculated the dynamics of pile-up formation against an obstacle. Screw dislocations were assumed to be in an inverse pile-up configuration at time $t=0$. A semi-logarithmic stress dependence of the dislocation velocity was assumed. Calculations were performed under a constant applied stress. The leading dislocations were observed to approach close to their final positions quickly and then slowly move to their final positions. The trailing dislocations were found to be immobile initially, then accelerate and finally decelerate. Also the velocity at any distance from the source was
found to decrease continuously and the maximum velocity position continually moves away from the obstacle. Using the materials constants for Fe-3% Si at 77°K, they calculated that the time required to create a stress concentration one half of its equilibrium value at the obstacle is of the order of 1 minute. Times required to create a stress concentration 90% of the equilibrium value is of the order of hours. They also suggest that this whole process will be even more sluggish in a strain rate insensitive material.

Kanninen and Rosenfield[107] considered the generation of dislocations from a source and their piling up against an obstacle on the slip plane away from the source. The calculations were performed under a constant stress and a linear stress dependence of the velocity of a dislocation was assumed. In this case they observed that the lead dislocation moved with a velocity 1.33 times the velocity of an isolated dislocation until it reached closer to its equilibrium position. Since a linear stress dependence for velocity was assumed this implied that the effective stress on the lead dislocation was 1.33 times the applied stress. The velocity of the trailing dislocations was found to be less the velocity of an isolated dislocation. The inter-dislocation spacing of the moving dislocations was found to be constant over most of the distance and for much of the time required to form the pile-up. Thus the presence of the obstacle changed the dislocation distribution from an inverted pile-up. The stress exerted at the head of the pile-up built up slowly at first and rapidly as the lead dislocation reached near the obstacle. It was shown that the stress on the obstacle depended on the rate of operation of the source. Performing calculations using material parameters of copper they showed that the time for essential completion of pile-up formation was $5 \times 10^{-5}$ sec.
Rosenfield and Kanninen[108] performed single ended pile-up generation and formation against an obstacle. Here they assumed a power law for the stress dependence of the velocity of the dislocation (\(v=Ma^n\)). Calculations were performed under a constant stress condition. It was observed that the stress build up at the obstacle when a new dislocation was emitted at the source was not a function of m value; but the rate of source operation was drastically slowed down as the m value increased. This lead to large increase in the time required to form equilibrium pile-up configurations. Although the process of pile-up formation is slowed down dramatically, they showed that significant stresses can be built up at the obstacle by non-equilibrium arrays. Also the stresses built up by equilibrium pile-ups was found to be well above the yield strength of material. Hence, it was suggested that in real materials equilibrium pile-ups are never achieved.

Arsenault[109] calculated the conditions for a dynamic pile-up formation in a neutron irradiated material. The sample was assumed to have a random distribution of short-range barriers and a periodic distribution of long range barriers. Short-range barriers could be overcome by thermal activation and the long range barrier was assumed to have a sinusoidal form and cannot be overcome by thermal activation. It was assumed that the short-range barriers can be removed once the dislocation overcomes the barrier. He showed that if a moving dislocation removed some percentage of the short-range barrier to the dislocation motion, dynamic pile-ups will form. Depending on the rate of removal they showed that three kinds of behavior are possible. First possibility is that the dynamic pile-up forms after some dislocations have exited the sample. In this the case the activation energies for the slip process will be that for the motion of a single dislocation. They showed that even in this case a dynamic pile will form and move at the speed of sound. The second possibility was that the dynamic pile-up forms before the first
dislocation exits the sample. This lead to decreased activation energies because of the increased stress level at the tip of the pile-up. The reason that activation energy was not zero in this case was because the slip process is thermally activated for some distance from the source. This implied that the time required to form a dynamic pile-up is still temperature dependent. The third possibility was a transition from case one to two and in this case a discontinuity in the experimental activation enthalpy versus effective stress was predicted.

Gerstle and Dvorak[110] developed a pile-up model to simulate the behavior of metals exhibiting delayed yielding. They simulated the formation of a pile-up and its release at yield. A viscous obstacle was introduced as a step increase in the drag stress opposing the dislocation motion. Such an obstacle was thought to represent a relatively weak obstacle which can be overcome and partially removed at yield. The yield criterion was established in terms of the equality of the velocity of the two leading dislocations. They assumed an exponential law for the stress dependence of the dislocation velocity. Both constant strain rate and creep conditions were simulated. The obstacle represented by the pinning drag stress and the source operation stress were observed to have little influence on the yield behavior, provided the obstacle drag stress was at least 1.2 times the drag stress experienced by the dislocations before reaching the obstacle and the applied stress was at least 1.1 times the source operation stress. The two quantities were kept constant in the simulations. This behavior was interpreted as the yield behavior being not determined by the obstacle strength or the nucleation of dislocations rather by the rapidity with which the dislocations can respond to the applied stress. The two important variables that does affect the yield behavior was determined to be the drag stress on the dislocations and the limiting velocity of the dislocations. In the case of creep calculations,
the lead dislocation was observed to support the highest stress all times. Between 15 and 16 dislocations were generated by the source during pre-yield in all cases and the stress concentration at the tip of the pile-up was found to be around 3.5 times the applied stress. The average velocity of the pile-up was observed to decrease continuously before yielding. After yield the pile-up was released within a small fraction of the delay time and the average velocity of the pile-up was observed to be constant. The time to yield increased with decrease in the applied stress in a non linear fashion.

Olfe and Neuhaüser[92] in their work on Cu-Zn alloys showed that a quasi-stationary moving group of dislocations will be established when some destruction of friction stress occurs in the slip plane. The stronger this destruction with respect to the number of dislocations, stronger the pile-up will be. They also showed that external stress only determines the velocity of such a pile-up and the friction stress at the head of the pile-up can be higher than the applied stress, but the dislocation group can still move. It was argued that the dislocations at end of the pile-up assist the lead dislocations in overcoming the friction stresses. But the dynamics of the critical pile-up formation or the velocity and stress distribution among the dislocations forming the mobile slip band was not shown.

All the above indicates that dislocations in a slip band will be in an “inverse pile-up” configuration when emitted from a source. This configuration does not depend on the velocity law used for the stress dependence of the dislocation velocity nor on the friction stress opposing dislocation motion. All these factors will affect the dynamics of the pile-up formation, but not the configuration of the pile-up. It was shown in Arsenault[109] and Olfe and Neuhäuser[92] that dynamic pile-ups will form only when there is a
destruction of obstacles to dislocation motion in the slip plane, i.e. when there is glide plane softening. Since, glide plane softening does occur due to the destruction of SRO in alloys with SRO, there will be formation of dynamic pile-ups in these alloys. It has been argued by Olfe and Neuhäuser[92] that the trailing dislocations in the pile-ups will assist the motion of leading dislocations by their self-stresses and the pile-up can be mobile at stresses lower than the friction stress due to SRO.

2.6 Room temperature creep behavior of titanium alloys

Room temperature creep of commercially pure (CP) Ti was first observed by Adenstedt[3]. Since then a number investigations have been done on this phenomena[4-6, 8-16, 20, 111]. The creep process is of transient kind or "exhaustion" type, i.e. the creep rate continually decreases with time [3-7, 11-13, 15, 16, 111]. The creep strains produced can be significant and is observed even when the applied stresses are below the yield strength of the material.

There was some controversy in the literature regarding the stress level below which creep does not occur under ambient conditions. Reimann [14] performed dead load torsional creep on Ti-6-4 tubular samples at room temperature. He concluded that room temperature creep of titanium alloys does not take place below 85 % of the yield stress. This statement is not true, as will be shown below. Imam and Gilmore[8] have suggested some reasons for Reimann's inability to notice creep at stresses below 85 % of YS. They suggested that the strain resolution of Reimann's setup was not sufficient to detect the strains that are produced during creep at stresses lower then 85% of YS. The second reason was that enough time was not given by Reimann at the lower stresses to detect the creep strains. In fact creep seems to occur at stresses much below the yield strength(as
low as 60% of yield strength[15]).

2.6.1 Representation of the creep curve

It was generally found [5, 6, 8, 11-13, 15] that creep curves obtained at room temperature/low temperature in Ti alloys could be represented by the following power law

$$\varepsilon = At^n$$

where \(A\) and \(n\) are constants, \(t\) is the time and \(\varepsilon\) is the creep strain. This is illustrated in figure 2.15, where data from several workers are plotted. Time exponent \(n\) is generally observed to be \(\approx 0.2\). The constants \(A\) and \(n\) are dependent on the applied stress and the microstructure. In fact Drefahl et al[11] have observed this power law to be valid over 27 years in Ti-6-4. Imam and Gilmore[8] observed some deviation from the power law in their work on Ti-6-4. It was observed that at the same stress value the time exponent \(n\) changed to a higher value after some strain.

2.6.2 Correlation of yield strength and creep resistance

As a first approximation, one would expect that a material with higher strength would show better creep properties. The work of Thompson and Odegard[15] on Ti-5-2.5 suggested that this is indeed true. They tested this alloy under three different microstructural conditions and the microstructure with the highest strength showed the most creep resistance. This evidence was also supported in the work of Odegard and Thompson[12] on Ti-6-4. In this work, Ti-6-4 was tested in both the aged condition and in the 'as welded' condition. The 'as welded' condition showed poorer creep properties and it also had the lower strength of the two conditions. Wapniarsky et al[16] tested Ti-6-4 under creep at the same fraction of the yield strength at room temperature in three microstructural conditions. Their condition A which had a \(\beta\) anneal, consisted of
equiaxed α (15μm) with grain boundary β. Condition B had an α-β anneal and consisted of equiaxed α and transformed β as fine plates. Condition A had higher strength (862 MPa) than condition B (843 MPa), but clearly as shown in figure 2.16, condition A had much lower creep resistance than condition B. But the creep resistance could not always be correlated with the 0.2 yield strength of the alloy. Chen et al [5] and Miller et al [11] studied room temperature creep properties of an α/β alloy, Ti-6wt.% Al-2wt.% Nb-1wt.% Ta-0.8wt.% Mo (Ti-6211). Their goal was to simulate the weld microstructures through heat treatment and test their creep properties. Table 2.4b gives the tensile properties of the various samples that they tested. It should be noted that samples W1 and W3 have yield strength of almost equal magnitude 760 MPa and 757 MPa respectively. Figure 2.17 shows that sample W1 although little stronger than sample W3, has creep properties much worse than W3. All the above data clearly points to the fact that creep resistance in these materials cannot be judged from their yield strength data. And that microstructure plays an important role in determining the creep resistance.

2.6.3 Effect of microstructure on primary creep of Ti Alloys

Thompson and Odegard [15] were the first to attempt correlation of creep resistance with the microstructure of the material. They performed creep tests at room temperature on Ti-5-2.5. This material was tested in three microstructural conditions. Their bar stock had an equiaxed microstructure of α grains of about 50μm grain size. The α forged material consisted of equiaxed α grains with a grain size of 3μm. The β forged material consisted of massive martensite (α'), in which colonies of parallel plates were separated by high angle boundaries which were prior β boundaries. The bar stock had the lowest strength, 834 MPa, β forged material had higher strength, 897 MPa and the α forged material had
the highest strength of 921 MPa. Creep tests were performed at 60 %, 80% and 90 % of their respective yield strengths on all three materials. The α-forged material showed the best creep resistance and the bar stock the worst. This difference was more pronounced at the lowest stress level (60% YS). At the higher stresses the difference between the various microstructures was not significant. It should be pointed out that the sample with α' (β forged material) as the dominant phase in the microstructure did not exhibit the highest creep resistance.

Odegard and Thompson[12] did room temperature creep studies on Ti-6-4. Specimens were tested in the aged condition (sample was annealed at 922°C for 15 min and water quenched, it was subsequently aged at 542°C for 4h and air cooled) and in the 'as welded' condition. The solution treated condition consisted of primary α grains with very fine martensite(transformed β) and the microstructure was not completely recrystallized. The fusion zone of the weld consisted of coarse widmanstätten structure and the heat affected zone consisted of basket weave structure with some primary α grains. The aged sample had higher yield strength than the 'as welded' sample. As mentioned previously, the aged material had better creep resistance than the welded sample.

Wapniarsky et al[16] conducted creep tests on Ti-6-4 at room temperature. Three different microstructures were tested. Condition A had equiaxed α (15μm) with grain boundary β, condition B had duplex microstructure with equiaxed α(10μm) and transformed β grains and condition C had very fine elongated α and dispersed β. The α plates were 2μm wide and 80μm long. They tested the alloy at 85, 90, 95% of the YS for the each microstructural condition. Condition A was found to be the least creep resistant. It was concluded that this was due to the presence of large amounts of β along the grain
boundaries. The presence of $\beta$ was thought to cause extensive grain boundary sliding. No microstructural evidence was provided to confirm this suggestion by the authors. The difference in the creep strains between microstructural conditions B and C was very small under 90% and 95% of the yield stress levels.

Chakrabarti and Nichols[4] have done creep studies on cast Ti-6-4 alloys in the temperature range 394-727K. Tests were performed on two microstructures. Both had coarse widmanstätten structures with grain boundary $\alpha$. The difference between the two is the prior $\beta$ grain size. Chakrabarti and Nichols called them fine grained and coarse grained material referring to the prior $\beta$ grain size. They concluded the following from their creep test data. The saturation strain at the same temperature and stress level was higher for the fine grained material compared to the coarse grained material. It was suggested that the higher plastic strain on loading and during creep deformation in the fine grained material was due to contributions from grain boundary and colony boundary sliding. Attempt was made by Chakrabarti and Nichols to observe grain and colony boundary sliding during the creep process. To achieve this scratches were inscribed on the surface of the sample and these were observed during the creep test. Figure 2.18a shows grid distortions at colony boundaries and evidence of coarse slip is shown figure 2.18b. The slip lines were observed to traverse the whole colonies and even continue across colonies were observed to distort across a colony boundary (see figure 31b). It should be noted that these samples were deformed at a temperature of 450K. Even though these are relatively lower temperatures compared to conventional creep, grain boundary sliding was observed at these lower temperatures. It is not clear whether this kind of grain and colony boundary sliding occur at room temperature. No attempt was made in this work to quantify the contribution of these sliding to the total creep strain.
Miller et al.[11] and Chen et al.[5] conducted creep studies on Ti 6221, an α–β alloy at ambient and high temperatures. Only ambient temperature results will be discussed here. Table 2.4a gives the various microstructures of the different samples tested by Miller et al. As discussed above W1 clearly shows the worst creep properties (see figure 2.17). This had the 'as received' Widmanstätten α-β structure. Sample, W3, which exhibited the best creep properties, had a martensitic structure and sample W2, which was only marginally worse than W3, had a basket weave type structure.

The above discussion clearly points out the influence of microstructure on the observed room temperature creep in many different titanium alloys. The mechanisms proposed to explain the microstructural effects will be discussed in the next section.

2.6.4 Mechanisms of Primary Creep

Various mechanisms have been proposed for the room temperature creep behavior of the Ti alloys. A survey of the main arguments will be presented below. The main phenomenological reason proposed is the high strain rate sensitivity, $m$ of titanium alloys[9, 12] and essentially two microstructural reasons have been proposed, difficulty in dislocation source activation and dislocation slip length. The microstructural arguments will be presented first and then the phenomenological argument will be discussed.

Thompson and Odegard[15] based on their work on Ti-5-2.5, suggested that the variation in the creep properties between different microstructures is due to the ease of source operation. Hence, the number of "easy" sources available in a microstructure decides
whether that microstructure is creep resistant or not. These sources are thought to activate during creep and contribute some creep strain. After some time the source gets exhausted and with time progressively more difficult sources have to be activated to produce strain. This leads to less and less strain with time, eventually stopping the creep process. It was postulated that their "bar stock" had the easiest sources and the $\alpha$-forged material had the most difficult sources to operate. In order to explain the intermediate creep response of the $\beta$ forged material, they suggested that $\alpha'$-martensite boundaries acted as dislocation sources in this condition. TEM evidence was provided, where dislocations appeared to emerge from the lath boundaries in this microstructure. This was interpreted as leading relatively easier source activation in the $\beta$ forged material and hence, the intermediate creep response. To further support this theory, Odegard and Thompson[12] conducted creep tests on Ti-6-4 at 60% of yield strength (YS) on samples whose surfaces were chemically polished to remove machining damage. This was in addition to those samples which were tested as machined. On such chemically polished samples they found that the creep strains were smaller than the creep strains accumulated in unpolished samples. This difference was more prominent for the lower stress values (60% YS) and was absent at a higher stress of 70% of YS value. Further, to support the exhaustion theory, prestrained (to a small strain) samples were tested in creep. The creep resistance was found to improve at the lower stresses, but at the higher stresses larger amounts of pre-strains had to be imparted to get better creep resistance. Prestraining to strains less than some critical strain worsened the creep resistance. The critical strain required is not an accurately known quantity. Practically, there could be a problem with this kind approach (pre-straining) to improve the creep resistance. It has been shown by a many workers that reversal of stresses after straining in one direction led to much worse creep properties.[8, 9, 14, 17, 18]
The argument that primary creep is due to operation of dislocation sources with a distribution of energies stems from Cottrell's [112] development of exhaustion creep theory. Cottrell's original development of exhaustion theory led to a logarithmic creep law, which is not observed in Ti alloys. Further, Cottrell was unable to satisfactorily modify the theory to accommodate Andrade creep [113], which is essentially the same as the power law given in equation 2.2 with the exponent \( a \) being 0.3. The exhaustion theory assumes that the sources once activated contributes a certain amount of strain and then stops. Once stopped these sources cannot be operated again. This may not be a realistic assumption. The amount of strain contributed by a source in this theory is an unknown factor. The exhaustion theory predicts creep strains much lower than what is observed in titanium alloys. If one observes the slip traces produced on the sample surface, the exhaustion theory would suggest production of new slip traces as a function of time. Thompson and Odegard [115] did not study the slip traces to provide evidence for new source operation as a function of time to support their conclusion.

Miller et al [11] and Chen et al [5] tested Ti-6211 under different microstructural conditions. They suggested that the difference in creep response between the different microstructures under similar testing conditions was due the variations in "slip length", which are dictated by the spacing of potential slip barriers. The poor creep resistance of sample W1 (see figure 2.17) which had an Widmanstätten microstructure was attributed to long slip lengths in that microstructure. The \( \alpha \) and \( \beta \) laths in widmanstätten structure have a burgers orientation relationship, given by
\{110\}_\text{bcc} \parallel (0001)_{\text{hcp}}

<111>_{\text{bcc}} \parallel <11\overline{2}0>_{\text{hcp}}

Hence, it was suggested by Chen et al\cite{5} and Miller et al\cite{11} that slip can occur across the entire colony. The slip length for this microstructure was thought to be the size of the colony. In the basketweave microstructure, they argue that there are many interfaces with no burgers orientation relationship and this leads to a smaller slip length. A sample with this microstructure, W2, showed better creep properties. The same kind of argument was made for the martensitic structure (W3), and this showed the best creep properties. The slip length argument although a very strong one, does not give the complete picture regarding the mechanism that is causing this creep. If we compare these results with that of Thompson and Odegard\cite{15}, they found that a structure with smaller equiaxed $\alpha$ grains exhibited better creep properties than a martensitic $\alpha'$ structure. In fact, they argued that the interfaces between the martensite plates act as dislocation sources leading to relatively poorer creep response of the material with a martensitic microstructure, compared to the equiaxed $\alpha$ sample with small grain size. If the slip length argument is true, in the work Wapniarsky et al \cite{16} one would expect their condition C with elongated $\alpha$ grains of only $2\mu$m width to show much better creep resistance than condition B with equiaxed grains of $10\mu$m. But there was only a very small difference in the creep behavior of the two alloys as was shown in figure 2.16. If smaller slip length leads to better creep resistance, which is an argument similar to Hall-Petch effect, one would expect alloys with smaller slip lengths to show higher yield strengths compared to alloys with larger slip length. This was not observed by Miller et al\cite{11} as shown in table 2.4b. And recently, it was shown by Suri et al\cite{114} that slip transmission through an $\alpha/\beta$ interface is highly anisotropic for a prismatic $<a>$-type slip system. Only one of the three possible slip vectors had an easy transmission process. Otherwise slip transmission was
shown to be a relatively difficult process. These results show that it is not reasonable to assume that colony size is the slip length in the case of widmanstätten structures.

Although grain boundary and colony boundary sliding was observed by many researchers[4, 5, 11], no attempt was made in any of the work to ascertain whether these boundaries are weak in nature or is it due to severe stress concentrations that these boundaries are sliding. Williams and Lütjering[57] have shown that the stress concentration produced by the planar slip caused premature cracking and led to poor fatigue properties in titanium alloys. Planar slip was observed by Thompson and Odegard and Odegard and Thompson[12, 15] in their work on both Ti-5-2.5 and Ti-6-4 (in the α phase). Such planar slip was observed by both Miller et al[11] and Chakrabarti and Nichols[4] in the α phase. An approach similar to Williams and Lütjering[57] in understanding the influence of this planar slip on the creep behavior has not been undertaken in the literature.

The main phenomenological argument is that the poor creep room temperature resistance of titanium alloys is due to their large strain rate sensitivity. This was first suggested by Hatch et al[9] and was later extended by Odegard and Thompson[12]. To prove this argument, Odegard and Thompson performed tensile tests at different strain rates. They observed that the measured yield strengths decrease as the strain rate is lowered. This is shown in figure 2.19. The dotted curve in figure 2.19 is the conjectured stress-strain curve thought to occur during creep as the average creep rates are very small. There are a few problems with this argument. Table 2.5[115] shows the strain rate sensitivity of Ti-5-2.5 along with other common materials which do not exhibit this creep behavior. As one can see the strain rate sensitivity of Ti-5-2.5 is not abnormally large. Odegard and
Thompson did not measure the actual initial creep rates in their tests. Their first data point was acquired after times of the order of minutes and the initial transient was not measured. There is evidence that the initial creep rates are very high[4]. This doesn't agree with their strain rate sensitivity argument.

In summary, titanium alloys exhibit creep at room temperature and can accumulate significant creep strains. Further, this process occurs over very long times (over years) and at stress levels much below the macroscopic yield stress of the material. The microstructure of the alloy plays a significant role in the creep properties. The main phenomenological reason proposed is that the strain rate sensitivity of the materials is very high and hence, they creep at stresses lower than the macroscopic yield stress. Comparison of strain rate sensitivity of titanium alloys with other metals and alloys show that they do not have abnormally high strain rate sensitivity. Two main microstructural mechanisms have been proposed to explain the occurrence of primary creep. One is a dislocation source argument, where these sources are easier to operate under certain microstructural condition and not in others. The second argument was that microstructural slip length determines which microstructure is more creep resistant. Basketweave and martensitic structures were observed to be more creep resistant than the widmanstätten structure. Based on the slip length arguments it was stated, that since, burgers orientation because orientation relationship were valid for $\alpha$ and $\beta$ laths in a widmanstätten structure, the colony length was thought to the slip length. Where as such orientation relationships were not assumed to be present between the $\alpha/\beta$ laths in a basketweave structure and in the martensitic structure. Hence, the much smaller dimension, lath spacing was assumed to be slip length. This was thought to explain the much better creep resistance of the basketweave and martensitic microstructures. It was
discussed above that both microstructural reasons completely satisfactory in explaining the room temperature creep in titanium alloys.
References


Figure 2.1: Microstructure of CP titanium. (a) annealed for 1h at 700°C showing equiaxed \( \alpha \) grains. (b) quenched from \( \beta \)-phase field showing martensitic \( \alpha' \) structure. (c) air cooled from the \( \beta \)-phase field showing Widmannstätten plates of \( \alpha \). (d) near \( \alpha \) alloy IMI-685 air cooled from the \( \beta \)-phase field showing a basketweave microstructure[2].
Figure 2.2: Microstructures of Ti-6Al-4V in six representative metallurgical conditions[1].
Figure 2.3: Typical Widmanstätten microstructure in a two phase alloy. The white phase is the α phase and the dark phase is the β phase[1].

Figure 2.4: Important crystallographic planes and directions in a hcp structure[22].
Figure 2.5: a) Ti rich end of the Ti-Al phase diagram proposed by Blackburn[26]. Our composition of interest is Ti-6wt% Al or Ti-10at% Al. b) Similar composition range of the phase diagram determined by Nambhoodiri et al[27] using resistivity measurements along with TEM studies.

Figure 2.6: Flow stress as a function of temperature and aluminum content in polycrystalline Ti-Al alloys. Aluminum was thought to strengthen titanium in an athermal fashion by Rosenberg and Nix[44].
Figure 2.7: CRSS for prism slip as a function of aluminum content in dilute titanium-aluminum alloys. There is some softening due to addition of aluminum. This was attributed to scavenging of interstitials by the addition of aluminum. The plot also shows the temperature dependence of CRSS for each aluminum content[40].

Figure 2.8: Same data as shown figure 2.7 is plotted on a temperature axis to illustrate the temperature dependence of yield strength. There is a strong temperature dependence of CRSS. For those which exhibits alloy softening, this dependence is weak. This was also taken as evidence for the scavenging hypothesis[44].
Figure 2.9: Stress-strain plot of prismatic slip at room temperature as function of aluminum content. Strain softening was observed in the Ti-5.2at% Al alloy and this was attributed to destruction of SRO during deformation[44].

Figure 2.10: CRSS for basal and prism slip as function of aluminum content at 300K, taken from the work of Paton et al [54]. Aluminum seems to strengthen prism slip more rapidly than basal slip.
Figure 2.11: Dislocations structures after 4% plastic strain in Ti-Al alloys from the work of Blackburn and Williams[38]. a)CP-Ti, b)Ti-5at%Al, c)Ti-10at%Al and d) Ti-13.5at%Al. Slip transition from homogeneous to planar slip is observed as the aluminum content is increased. Deformation is very planar in both Ti-10at% Al and Ti-13.5 at% Al.
Figure 2.12: Schematic of the core structure proposed for \(<a>\) type screw dislocations in \(\alpha\)-Ti by Naka et al[63]. In this structure, the core of the screw dislocation is spread on three different planes, on the prism plane and two symmetrically oriented pyramidal planes. (a) shows the sessile configuration of the screw dislocation. (b) and (c) show the glissile configuration of the core structure on prism and pyramidal planes respectively.

Figure 2.13: Differential displacement plots showing the core structure of \(<a>\) type screw dislocations in \(\alpha\)-Ti on basal planes (a) and on prism planes (b). The dislocation core is spread on both prism and basal planes[66]. (continued)
Figure 2.13: continued.

Figure 2.14: Configurational energy change $\gamma_{\text{SRO}}$ in Ni-11.2at%Mo as function of number of dislocations(n) shearing the slip plane. It shows that the diffuse APB energy oscillates for the first 5 dislocations after which it is not altered by subsequent dislocations. This $\gamma_{\text{SRO}}$ is estimated as a function of temperature (corresponding to the SRO state at those temperatures) in steps of 100K, top 473K and bottom 1173K [83].
Figure 2.15: Log creep strain vs log creep time data for different titanium alloys with different microstructures. Titanium alloys seem to have a creep time exponent of 0.2 at long times.

Figure 2.16: Room temperature creep data of Ti-6Al-4V, tested at 90% of the yield strength (a) and at 95% of the yield strength (b). Even though condition A had a higher yield strength (862 MPa) compared to condition B (843 MPa), it is clear that the alloy in condition A is much less creep resistant[16].
Figure 2.17: Room temperature creep in a Ti-6211 alloy as function of microstructure. See table 2.4a for the sample designations. All samples were tested at the same applied stress level of 684 MPa. Even though sample W1 and W3 have comparable yield strengths (see table 2.4b), W1 has much lower creep resistance[11].

Figure 2.18: Surface slip line observations and fiducial grid distortion in a Ti-6Al-4V sample. In (a) colony boundary sliding can be noticed from the grid distortion (d) and in (b) relative displacement between grains($d_g$) and coarse slip lines can be seen. The sample was deformed at 450K to a creep strain of 0.85%[4].
Figure 2.19: Comparison of tensile test results at various strain rates with the creep results. Creep strains are from 1000h creep tests and the dashed line is a conjectured line [12]. Since the yield strength of the material decreases as the strain rate is lowered, it was implied by Odegard and Thompson that under creep conditions the strain rates are so low that, it is equivalent to testing the alloy at this very low strain rate. Hence, it was suggested that the applied stress level might be above the yield strength at such low strain rates.
<table>
<thead>
<tr>
<th>Metal</th>
<th>c/a</th>
<th>Packing densities</th>
<th>Observed slip planes in order of ease of operation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cadmium</td>
<td>1.886</td>
<td>1.000 0.918 0.816</td>
<td>(0001) (10T0) (10T1)</td>
</tr>
<tr>
<td>Zinc</td>
<td>1.856</td>
<td>1.000 0.933 0.846</td>
<td>(0001) (1100) (11T2)</td>
</tr>
<tr>
<td>Magnesium</td>
<td>1.624</td>
<td>1.000 0.940 0.940</td>
<td>(1000) (1011) (1100)</td>
</tr>
<tr>
<td>Titanium</td>
<td>1.587</td>
<td>1.000 1.092 0.959</td>
<td>(1100) (0001) (1011)</td>
</tr>
</tbody>
</table>

Table 2.1: Atomic packing densities of various planes in hcp systems with different c/a ratios[23].

<table>
<thead>
<tr>
<th>Composition</th>
<th>Strain Coefficient, A. Fum</th>
<th>Strain Exponent</th>
<th>Strain at Maximum Load, E%, No. per En.</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 Ti</td>
<td>0.44</td>
<td>0.25</td>
<td>0.30</td>
</tr>
<tr>
<td>1 Al</td>
<td>2 Al</td>
<td>2.9 Al</td>
<td>6.33</td>
</tr>
<tr>
<td>3 Al</td>
<td>4 Al</td>
<td>7.5 Al</td>
<td>6.33</td>
</tr>
</tbody>
</table>

Figure 2.2: Strain hardening exponent as a function of aluminum content in Ti-Al alloys upto 7wt%Al. from the work of Ogden et al[43].

<table>
<thead>
<tr>
<th>Al content (at%)</th>
<th>$\frac{\tau_{basal}}{\tau_{ prism}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.44</td>
<td>5.34</td>
</tr>
<tr>
<td>0.87</td>
<td>6.85</td>
</tr>
<tr>
<td>1.2</td>
<td>6.33</td>
</tr>
<tr>
<td>2.11</td>
<td>4.43</td>
</tr>
<tr>
<td>5.2</td>
<td>2.43</td>
</tr>
</tbody>
</table>

Table 2.3: Ratio of CRSS for basal slip versus prism slip as function of aluminum content at 298K taken from the work of Sakai and Fine[51]. It shows that the CRSS ratio decreases with increasing aluminum content.
Table 2.4: List of heat treatments, sample designations and microstructure are listed in (a) and the mechanical properties are listed in (b) for Ti-6211 alloy. Both taken from the work of Miller et al [11].
Table 2.5: Comparison of the strain rate sensitivity of a titanium alloy, Ti-5Al-2.5Sn with other conventional materials, taken from the work of Read[115]. It is clear that titanium alloys do not have abnormally high strain rate sensitivity compared to other conventional metals and alloys.
CHAPTER 3

EXPERIMENTAL PROCEDURES

3.1 Material history: processing and heat treatments

3.1.1 Processing History

The Ti-6wt% Al alloy (Ti-6Al), a single phase α (hcp) titanium alloy that was used for the majority of the study was obtained from two different sources. One batch of material was obtained from Duriron company, Dayton, Ohio as 12.7 mm diameter cast rods. These samples have an average grain/variant size of approximately 500µm. The nominal composition of the as received material provided by Duriron is listed in table 3.1. The samples were annealed at 900°C for 5 hr and furnace cooled before testing. The second batch of material was obtained from RMI company, Niles, Ohio. These were obtained as 12.7mm rods. The alloy was first cast as 250g buttons and were then subjected to thermo-mechanical processing to produce the rods. The processing sequence is listed in table 3.2. All the samples were annealed at 900°C for 24 hours before various other treatments. The various heat treatments that were performed for all mechanical property studies (creep,
constant strain rate and in-situ deformation studies) are listed in table 3.3 and those used in neutron diffraction studies are listed table 3.4. The samples had an average grain size of approximately 75 microns after the annealing treatment. Polycrystalline Ti-6242 material were machined from a ring forging made by RMI titanium company and have a two phase $\alpha/\beta$ equiaxed structure with an average grain size of approximately 20$\mu$m. The heat treatment of Ti-6242 consisted of an 1 hour anneal at 927°C followed by an air cool. The material was then subjected to a stabilization treatment of 8 hours at 593°C and air cooled. These heat treatments were performed at Metals Technology Inc.

3.1.2 Heat treatment procedure

Samples were heat treated in sealed quartz tubes back filled with high purity argon. The samples were placed in cleaned quartz tubes (ID=14mm, OD=16mm, L=203mm) along with high purity titanium getter strips and evacuated to a vacuum of better than $4 \times 10^{-3}$ Pa using a oil diffusion pump. The tubes were then backfilled with high purity argon to a pressure of $<1000$ Pa and sealed. Finally, one of the getter strips was heated using acetylene torch, while keeping the sample cool by wrapping that portion of the tube with a napkin soaked in water, to remove any residual interstitial impurities present in the sealed quartz tube. The samples were then placed in a tube furnace which had already reached the heat treatment temperature. A thermocouple was placed right next to the sample to ensure that the sample experienced the correct temperature.
For the ice water quench treatments, additional weight (Ti-6Al rod) were placed in the tubes separated from the sample by a getter strip. The quartz tubes also had hooks to which wires were attached which facilitated faster sample removal from the tube furnace. After the annealing treatment the sample was quickly pulled out of the furnace and quenched in an ice water bath at 0°C and the tube was broken immediately. Additional weight that was placed in the tubes helped the sample to sink in the bath, thus ensuring an uniform quench.

For the step quenching heat treatments, two furnaces were heated to the desired temperatures. The initial high temperature anneal was done in a box furnace with the thermocouple next to the sample. After this treatment the sample was transferred immediately from the box furnace to another tube furnace which was held at the ageing temperature. This whole transfer process was achieved within 1-2 minutes.

3.2 Mechanical Testing

3.2.1 Compression Testing

The compression samples were 4mmx4mmx12mm parallelepipeds which were machined with all surfaces ground to 600 grit. All constant strain rate and creep tests were performed in a compression cage. The strains were measured using a strain gauge mounted directly on one sample face. The approximate strain resolution of the strain gauge was 5x10^-6. The strain and load data were acquired using a high-speed LabView computer-based data acquisition system. The constant strain rate tests were done in an electro-mechanical, screw-driven Instron 1362 machine. The strain rate was controlled during the test using an LVDT mounted on rod and tube extensometry across the
compression cage. The strain rate sensitivity was measured from strain rate jump tests. For the creep tests, the samples were tested in a dead load ATS creep frame with a 20:1 lever arm ratio. Figure 3.1 shows the creep frame and the LabView computer-based data acquisition system used.

3.2.2 Tensile Testing

Constant strain rate and creep tests were also conducted in tension in both Ti-6Al and Ti-6242. Standard tensile test specimens according to ASTM-E8M specifications for small size specimens were used. The samples had a diameter of 6mm and a gage length of 30mm. These tests were conducted at Metals technology Inc., Northridge, CA. Constant strain rate tests were conducted in a Tinius-Olsen screw driven machine with a model CMH 289 interface controller. Strains were measured by attaching a Tinius-Olsen extensometer to the gage section of the sample. Creep tests were conducted in a Satec systems M3 constant load creep frames with a Satec systems computer-based data acquisition system. Creep strains were measured using Satec systems dual LVDTs.

3.3 Slip line evolution studies

Creep tests were done where the test was interrupted periodically to study the slip line evolution during creep. For this purpose one of the sample faces was polished for optical metallography. The sample was taken out after complete unloading and pictures were taken using an optical microscope. After capturing the slip lines on the polished surface, the samples were reloaded to their previous load and the test was continued.
3.4 Sample Preparation for Optical Metallography

Samples for optical metallography were cut either in a Struers Accutom-50 saw at 3000 rpm using silicon carbide blades or in Leco high speed saw using silicon carbide blades. They were then mounted using Sample-Kwick cold mount epoxy or bakelite. The samples were ground in silicon carbide paper from 400 grit to 1200 grit at 150 to 200 rpm in running water. The fine polishing was done in a Syntron vibratory polisher using 0.3 micron alumina slurry on a Beuhler microcloth. After polishing, the samples were etched with Kroll’s etch and imaged using polarized light or were etched with Weck’s tint etch and imaged in non-polarized light.

3.5 Sample Preparation for Transmission Electron Microscopy

After mechanical testing, discs for transmission electron microscopy (TEM) investigation were cut using Struers Accutom-50 saw at a speed of 300rpm, using low force and 0.015inch thick, 4 inch diameter silicon carbide blade. Discs were usually cut to a thickness of 275 to 325 microns. They were mechanically ground in 400grit and then 600 grit paper to 200 microns. After this, 3mm diameter discs were cut from the 4mm square discs using a South Bay slurry drill using consumable brass drill bits. The 3mm discs were ground to a thickness of 170 to 180 microns using 600 grit to 1200 grit paper. Finally, thin foils were prepared by electropolishing in a Streurs Tenupol-3 unit. The polishing solution used was 6% perchloric acid, 35% 2-butoxy ethanol and 59% methanol. The samples were polished in the temperature range of -40°C to -30°C at a voltage of 10 V.
3.6 Transmission Electron Microscopy

TEM investigations involved conventional g.b analysis of dislocations as well as imaging faults. Both bright field and dark field imaging were employed in the investigations. These investigations were performed using a Philips CM200 electron microscope (with a LaB₆ cathode) under an operating voltage of 200kV. Some images were acquired using a CCD camera attached to the Gatan Imaging Filter system (GIF) in a Philips CM200 microscope. The filtered images were acquired at a voltage of 197kV using zero loss electrons within a window of 20eV.

3.7 Neutron diffraction studies

Samples for neutron diffraction studies were sealed and heat treated in quartz tubes in the same fashion as described in section 3.1.2. Cylinders of 10mm diameter and approximately 84mm in length were used for these studies. Neutron diffraction experiments were conducted in Oak Ridge National Labs (ORNL) at the High Flux Isotope Reactor facility (HFIR). These experiments were conducted under the guidance of Dr. J. Lee Robertson at ORNL. Neutron powder diffraction patterns were collected from polycrystalline Ti-6Al specimens. The sample was spun during the experiment to minimize texture effects. Experiments were conducted in the HB1A Triple-Axis Spectrometer with neutrons of wavelength 2.357Å (E = 14.8meV). The incident beam monochromator system consisted of a vertically focusing graphite (PG) monochromator crystal at the main beam and second vertically focusing graphite (PG) monochromator at a second position. The main monochromatic beam was passed through a slit to the sample. Diffracted beam from the sample was collimated through a 40° collimator to the graphite analyzer crystal and through a second 138° collimator to the detector. This set up
is schematically illustrated in figure 3.2. The collected intensity was corrected for both air scattering, background scattering and also corrected for absorption inside the sample.

3.8 In-situ deformation studies in a HVEM

Several samples were deformed in-situ in a high voltage electron microscope (HVEM) to study the dislocations processes. These experiments were performed in collaboration with Prof. U. Messerschmidt and Dr. Martin Bartsch at the Max Planck Institute for Microstructure Physics in Halle/Saale, Germany. For the in-situ deformation studies, specimen blanks of 2-2.4mm wide, 8mm long and 0.1mm thick were cut using Struers Accutom-50 saw after various heat treatments. These samples were then electro-polished in two stages using a Struers Tenupol-3 twin-jet polisher. The polishing solution was the same that was described in section 3.5. A mask was put in and the central portion of the samples were thinned first. In the second step the mask was taken out and the whole sample was thinned. This two step process ensured that the thinnest region of the samples were in the center of the foil. The sample were then glued onto the in-situ stage using a cyanoacrylate resin. Samples were deformed in tension at room temperature in a JEOL HVEM operating at 1MeV. For an interesting history of this 25 year old microscope see Kästner and Messerschmidt[1]. Details about the straining stage can be obtained from the work of Messerschmidt and Appel[2]. During in-situ deformation several micrographs were taken as well as the deformation was captured in a video cassette.

3.9 In-situ heating studies in a TEM

In-situ heating studies were conducted to characterize weak-fringing faults observed in titanium alloys. For this the sample was heated in-situ in a Philips CM200 electron
microscope using a Gatan model 652 double tilting heating holder. Figure 3.3 shows a drawing of the double tilt holder. The sample was resistively heated and the holder was water cooled.
References


Figure 3.1: In (a) a dead-load ATS creep frame with a 20:1 lever arm ratio is shown. The hydraulic lift used for loading can also be seen. LabView computer-based data acquisition system is shown in (b).

Figure 3.2: Schematic of the HB1A triple axis spectrometer set up used for neutron diffraction studies at ORNL.
Figure 3.3: (a) Model 652 Gatan double tile heating holder on stand. (b) Specimen tip of the holder. This holder was used for in-situ heating of samples in a Philips CM200 microscope to study the effect of heating on the weak fringing fault contrast. These studies are described in chapter 7.
### Table 3.1: Nominal chemical composition of as received Ti-6Al material.

<table>
<thead>
<tr>
<th>Source</th>
<th>Al (wt%)</th>
<th>Fe (wt%)</th>
<th>O (wt%)</th>
<th>N (wt %)</th>
<th>C (wt%)</th>
<th>H (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duriron</td>
<td>6.5</td>
<td>0.06</td>
<td>0.08</td>
<td>0.01 (wt ppm)</td>
<td>0.01</td>
<td>0.003</td>
</tr>
<tr>
<td>RMI-1</td>
<td>6.28</td>
<td>0.014</td>
<td>0.042</td>
<td>0.002</td>
<td>0.008</td>
<td></td>
</tr>
<tr>
<td>RMI-2</td>
<td>5.94</td>
<td>0.036</td>
<td>0.042</td>
<td>0.004</td>
<td>0.007</td>
<td></td>
</tr>
<tr>
<td>RMI-3</td>
<td>5.84</td>
<td>0.032</td>
<td>0.043</td>
<td>0.002</td>
<td>0.008</td>
<td></td>
</tr>
<tr>
<td>RMI-4</td>
<td>6.03</td>
<td>0.015</td>
<td>0.042</td>
<td>0.004</td>
<td>0.015</td>
<td></td>
</tr>
</tbody>
</table>

### Table 3.2: Thermo-mechanical processing history of Ti6Al-RMI material

<table>
<thead>
<tr>
<th>Step Number</th>
<th>Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Heat to 1149°C and forge to 7/8 inch diameter x length</td>
</tr>
<tr>
<td>2</td>
<td>Heat to 954°C and roll to 5/8 inch diameter x length</td>
</tr>
<tr>
<td>3</td>
<td>Heat to 954°C and roll to 1/2 inch diameter x length</td>
</tr>
<tr>
<td>Source</td>
<td>Heat treatment</td>
</tr>
<tr>
<td>---------</td>
<td>-------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Duriron</td>
<td>900°C-5h anneal-furnace cool</td>
</tr>
<tr>
<td>RMI-1</td>
<td>A-ice water quench(IWQ)</td>
</tr>
<tr>
<td>RMI-1</td>
<td>A-IWQ to room temperature(RT) and aged at Y°C for X hours and IWQ to RT</td>
</tr>
<tr>
<td>RMI-1</td>
<td>A-air cool</td>
</tr>
<tr>
<td>RMI-1</td>
<td>A-air cool to RT and aged at Y°C for X hours and IWQ to RT</td>
</tr>
<tr>
<td>RMI-1</td>
<td>A and step quenched to the ageing temperature(Y°C) and aged for X hours and IWQ to RT</td>
</tr>
<tr>
<td>RMI-1</td>
<td>A-step quenched to 600°C and aged for 24h and IWQ to RT and aged again at 400°C for 5h and IWQ to RT.</td>
</tr>
</tbody>
</table>

Table 3.3: List of sample labels for Ti-6Al alloy with various heat treatments used in the study of mechanical properties. All RMI samples were annealed for 24h at 900°C (labeled A) before they were cooled at different rates and aged at various temperatures for different times.
<table>
<thead>
<tr>
<th>Source</th>
<th>Heat treatment</th>
<th>Label</th>
</tr>
</thead>
<tbody>
<tr>
<td>RMI-2</td>
<td>A-ice water quench(IWQ)</td>
<td>IWQ</td>
</tr>
<tr>
<td>RMI-2</td>
<td>A-IWQ to room temperature(RT) and aged at 500°C for 1 hour and IWQ to RT</td>
<td>IWQ-500C-1h</td>
</tr>
<tr>
<td>RMI-2</td>
<td>A-IWQ to RT and aged at 300°C for 100 hours and IWQ to RT</td>
<td>IWQ-350C-100h</td>
</tr>
<tr>
<td>RMI-4</td>
<td>A-IWQ to RT and aged at 650°C for 24 hours and IWQ to RT</td>
<td>IWQ-650C-24h</td>
</tr>
<tr>
<td>RMI-4</td>
<td>A and step quenched to the ageing temperature, 650°C and aged for 24 hours and IWQ to RT</td>
<td>SQ-650C-24h</td>
</tr>
<tr>
<td>RMI-3</td>
<td>A-air cool</td>
<td>AC</td>
</tr>
<tr>
<td>RMI-3</td>
<td>A-air cool to RT and aged at 400°C for 5 hours and IWQ to RT</td>
<td>AC-400C-5h</td>
</tr>
<tr>
<td>RMI-4</td>
<td>A and step quenched to the ageing temperature, 600°C and aged for 24 hours and IWQ to RT</td>
<td>SQ-600C-24h</td>
</tr>
<tr>
<td>RMI-2</td>
<td>A-step quenched to 600°C and aged for 24h and IWQ to RT and aged again at 400°C for 5h and IWQ to RT</td>
<td>SQ-600C-24h-400C-5h</td>
</tr>
</tbody>
</table>

Table 3.4: Identification of samples used in neutron diffraction studies.
CHAPTER 4

CHAPTER 4

CHARACTERIZATION OF SHORT-RANGE ORDER (SRO) USING NEUTRON DIFFRACTION

4.1 Introduction

Based on deformation studies, Blackburn and Williams[1] first suggested that there is short-range order (SRO) of titanium and aluminum atoms in binary Ti-Al alloys with Al content <6wt% Al. They observed a slip transition from homogeneous to planar slip with increasing aluminum content. In a Ti-6wt% Al alloy they observed planar slip and pairing of lead dislocations, but did not observe any $\alpha_2$(Ti$_3$Al) particles. Further, they noted that the increase in yield strength with aluminum concentration had a $C^2$ dependence in alloys containing up to 6wt% Al, and the concentration dependence of yield strength for higher aluminum alloys was weaker. In the higher aluminum alloys they observed precipitation of $\alpha_2$ particles. Based on the above they suggested that the occurrence of planar slip in alloys containing 6wt% Al or less is due to destruction of SRO and not due to a decrease in stacking fault energy with the addition of aluminum. Namboodhiri et al[2] studied the Ti-rich end of Ti-Al phase diagram using both electrical resistivity and transmission electron microscopy (TEM). They conducted isothermal ageing experiments (after solution treatment) from 450°C and above. In a Ti-3.9wt% Al alloy they observed a
maxima in resistivity when aged at 450°C for ≈30h. Similarly, in a Ti-6wt% Al alloy they observed resistivity maxima after ageing for 6h at 505°C and after ageing for 3h at 550°C. In this alloy they observed α2 superlattice reflections in electron diffraction patterns only after ageing for 400h at 505°C. Further, in their studies of a Ti-7.7wt%Al alloy, the resistivity maxima coincided with the precipitation of the α2 phase, when aged for 40h at 505°C. Since, the resistivity maxima occurred at earlier times in both Ti-6wt% Al and Ti-3.9wt% Al compared to the Ti-7.7wt% Al, they concluded that the peak in resistivity was due to the presence of SRO. Thus, in the literature indirect evidence has been provided for the presence of SRO in dilute Ti-Al alloys, but there are no direct diffraction studies to confirm the presence of SRO in these alloys. In this chapter, neutron diffraction results will be presented which will show direct evidence for the presence of SRO in a Ti-6wt% Al alloy (Ti-6Al) after different heat treatments. Such studies have been conducted on samples which were heat treated to modify the SRO state. Room temperature creep studies were conducted on samples with similar heat treatments, the results of which are discussed in chapter 6.

4.2 Background

As discussed before, in the literature, there is no diffraction evidence for the presence of SRO in Ti-Al alloys. In the studies of both Blackburn and Williams[1] and Namboodhiri et al[2] electron diffraction did not indicate the presence of SRO. This is true in our studies as well. There was no evidence for diffuse peaks at the superlattice positions corresponding to the α2 structure in electron diffraction. In order to observe such diffuse peaks, the difference between the atomic scattering factors for electrons for titanium and aluminum atoms must be large. In figure 4.1 atomic scattering factors of titanium and aluminum is plotted as a function of sin(θ)/λ for both electrons[3] and x-rays[4]. The plot
shows that the atomic scattering factor difference is relatively small. Therefore, it is
difficult to observe the diffuse peak due to SRO above the background because the peak
intensity will be very low for both x-rays and electrons. The scattering factors for neutron
diffraction are also listed in figure 4.1. Since, the scattering factor for titanium is negative
and that for aluminum is positive[5], diffuse peaks due to SRO will be easier to detect
using neutrons.

4.3 Short-range order in Ti-6Al

A typical neutron diffraction “powder” pattern obtained from Ti-6Al is shown in figure
4.2. The incident neutrons had a wavelength of 2.357Å. Various hcp peaks are labeled in
figure 4.2. The main diffuse peak due to SRO is between 20° to 50° in 20 and this has
been identified as DP. This 20 range is magnified in figure 4.3 and one can observe the
diffuse peak clearly. However, the diffuse peak is very weak in this sample because it has
been ice water quenched after an anneal at 900°C to minimize the SRO. The positions of
the superlattice peaks due to α2 are also labeled in figure 4.3. The maxima in the diffuse
peak is around 20=40° which corresponds to the position of the {1011} reflection of α2
which should have the maximum intensity among three α2 reflections listed in the
figure(indicated by the heights of the lines). This confirms that the SRO present in Ti-6Al
is related to the ordered α2 phase and can be thought of as a precursor to the ordered
phase. Another point to be noted is that the position of the diffuse peak is about 1°-2° to
the left of the position of the {1011} reflection whose position has been calculated
assuming the lattice parameters of the ordered α2 phase. This can be clearly seen in figure
4.4.
Several heat treatments were done to modify the SRO state. These heat treatments and the sample labels are listed in table 3.4. Creep studies were conducted after these treatments which are discussed in chapter 6. Neutron diffraction studies were conducted in Ti-6Al after many of these heat treatments to characterize the SRO state. Figure 4.4 compares the diffuse peak magnitude between an IWQ sample and a IWQ-500C-1h sample. As discussed in section 2.3, the $\alpha/\alpha_2+\alpha$ phase boundary lies at $\approx 550^\circ$C for Ti-6Al[2, 6]. Hence, the second sample was aged inside the two phase region for a short time. It is apparent that the magnitude of the diffuse peak is much stronger than the one after an IWQ treatment. This shows that the SRO state has been modified after such a treatment. It should be pointed out that when a sample was aged for 24h at $350^\circ$C after the IWQ treatment, there was no significant difference in the diffuse peak between this and the IWQ sample. This indicates that significant change in the SRO state has not occurred in this sample. Longer ageing times at this temperature or higher ageing temperatures are required to modify the SRO state.

Figure 4.5 compares the diffuse peaks in a IWQ-650C-24h sample and a SQ-650C-24h sample. Both samples were aged at the same temperature, $650^\circ$C for 24h after a high temperature anneal at $900^\circ$C for 24h. The difference between the two is that in the first case the sample was IWQ to room temperature and then aged and in the second case the sample was quenched directly to the ageing temperature. Essentially there is no difference between the two peaks, although it appears as though the IWQ-650C-24h sample has slightly higher intensities. But the whole peak of the IWQ-650C-24h sample lies above the SQ-650C-24h sample. This could be due to slightly different sample diameters between the two samples. Practically, there is no difference between the two peaks. Therefore, the SRO state in the two samples are very similar, which is reasonable.
because both samples were aged above the $\alpha/\alpha_2+\alpha$ transus at relatively high temperatures. The corresponding creep behavior was similar for both samples as discussed in chapter 6 (see figure 6.6).

But clear correlation between the SRO state and the creep behavior was not always possible. Figure 4.6 compares the SRO peak between an AC sample and an AC-400C-5h sample. Clearly, there is a significant difference in the diffuse peak between the two samples. The magnitude of the diffuse peak is stronger in the AC-400C-5h sample. The overall creep response was not very different between the two samples, but there was a significant difference in the initial transient (see figure 6.5). In the case of SQ-600C-24h and SQ-600C-24h-400C-5h samples the opposite seems to be true. The SRO peaks from these two samples are compared in figure 4.7. The peak after the SQ-600C-24h-400C-5h treatment is slightly stronger than the SQ-600C-24h treatment. But this difference is smaller than that was observed between the AC and AC-400C-5h samples. The creep response of the SQ-600C-24h-400C-5h sample was very different from that of the SQ-600C-24h sample. The former sample was much stronger in creep (see figure 6.7).

Finally, SRO peaks are compared between samples that have undergone many different ageing treatments to modify the SRO state in figure 4.8. The differences in the peak shape and magnitude among the different samples is relatively small. But there are significant differences in the creep responses (discussed chapter 6). Therefore, direct correlation between the creep response and the SRO diffuse peak is very difficult at this point in these aged samples. Calculation of Warren-Cowley short-range order parameters from the diffuse peaks will provide a more complete characterization of the SRO state of the various samples. There could be subtle differences in the SRO state between the various samples which could be revealed by such an analysis. A straightforward
correlation between the SRO state and the creep response might be possible after such
detailed analysis of the data. These analyses are being carried out now.

4.4 Summary

a) Direct diffraction evidence for the presence short-range order of titanium and
aluminum atoms in a Ti-6Al alloy has been provided for the first time. The position of
the diffuse peak indicates that the SRO state is related to the ordered \( \alpha_2 \) phase, \( Ti_3Al \) with
\( DO_{19} \) structure. It can be thought of as a precursor to the \( \alpha_2 \) phase.

b) SRO diffuse peak was observed to be very weak in the IWQ sample. It was shown that
the SRO state can be modified by subsequent ageing treatments. There is significant SRO
after a conventional treatment like an air cool in these alloys.

c) Direct correlation between SRO diffuse peak magnitude and creep response of samples
(which is discussed in chapter 6) was possible for the IWQ, IWQ-650C-24h and SQ-
650C-24h samples. Clear correlation between creep response and SRO state was not
straight forward for samples that were aged to produce stronger SRO.
References


Figure 4.1: Atomic scattering factors of titanium and aluminum for x-rays, electrons and neutrons are shown above. One can observe that the difference in scattering factors is relatively small for electrons and x-rays, where as for neutrons the scattering factor of titanium is negative. Hence, it is easier to detect the diffusing scattering due to SRO of titanium and aluminum atoms using neutrons.

Figure 4.2: A typical neutron diffraction pattern from polycrystalline Ti-6Al. Diffraction peaks due to disordered hcp structure are labelled. The main diffuse peak due to SRO is indicated as DP. Incident thermal neutrons had a wavelength of 2.357Å.
Figure 4.3: Magnified view of the diffuse peak indicated in figure 4.2 in an IWQ sample. Only the diffuse peak is shown and the line is fit through the diffuse peak only. This is true for all the following figures as well. The super lattice peaks that would be present due to ordered Ti₅Al with DO₁₉ structure is also indicated. The diffuse peak is relatively weak in this IWQ sample.

Figure 4.4: Comparison of SRO diffuse peak between the IWQ sample and an IWQ-500C-1h sample. The peak appears to be stronger in the case of the aged sample. This shows that the SRO state is modified by subsequent ageing treatments.
Figure 4.5: SRO diffuse peak comparison between two samples, an IWQ-650C-24h and a SQ-650C-24h sample. The diffuse peak is very similar between the two samples, indicating that the SRO state could be similar in both samples. Creep studies discussed in chapter 6 indicated the same.

Figure 4.6: Diffuse peak present in an AC sample is compared with a sample that was aged at 400°C for 5h after the AC treatment. The SRO diffuse peak appears to be stronger in the case of the aged sample. This would indicate a stronger SRO state in the aged sample. But, differences in the overall creep transients (discussed in chapter 6) were small.
Figure 4.7: Here the SRO state between a SQ-600C-24h sample and SQ-600C-24h-400C-5h sample is compared. There is a small difference in the magnitude of the diffuse peak, with SQ-600C-24h-400C-5h sample having a marginally stronger peak. This difference is smaller than what was observed between the AC and AC-400C-5h samples (figure 4.6). As shown in chapter 6, the creep behavior of the two SQ samples is very different.

Figure 4.8: SRO peak intensities of many samples with various heat treatments are shown here. From the plot it is clear that the difference in the SRO between the different ageing treatments is subtle and peak intensities fall in a narrow band.
CHAPTER 5

PHENOMENOLOGICAL ANALYSIS OF PRIMARY CREEP

5.1 Introduction

During low temperature creep (<0.25T_m) of many metals and alloys, primary creep is the dominant deformation mode. At low creep stresses and creep strains (<2x10^{-3})[1], the primary creep deformation of many metals and alloys has been described by a logarithmic creep law of the form \( \varepsilon = \alpha \ln t + C \) [1]. In cases where there is larger accumulation of primary creep strains, the deformation often can be described by a power law function of creep strain with time:

\[ \varepsilon = At^a \]  \hspace{1cm} (5.1)

where \( A \) and \( a \) are constants. The time exponent \( a \) indicates the exhaustion rate during creep. Smaller \( a \) values indicate more rapid exhaustion during creep, with \( a=1/3 \) being the classic Andrade creep[2]. Recently, Cottrell[3-5] and Nabarro[6] have revisited the issue of Andrade creep and have proposed dislocation-based models to explain the Andrade creep exponent of \( a=1/3 \). However, from the low temperature creep data to be found in the literature[1, 7, 8], it is clear that a creep time exponent of 1/3 is not unique and is found to vary widely from 0.03 to 1 for different materials[1]. This is also the case for the titanium alloys to be described below.

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In titanium alloys, even for stresses well below the yield strength, significant strain can accumulate during creep as a function of time at room temperature. In polycrystals the primary creep transient is of the exhaustion type, and can generally be described by the power-law of the form shown in equation 5.1. Steady state creep is generally not observed in these alloys at room temperature. It has been observed that the constant $A$ depends upon both microstructure and stress. For a compilation of creep data from several groups\[9-11\] where the creep strain versus time is plotted on log-log axes, see figure 2.15. Most of the creep data for titanium alloys seem to be linear on this plot with a time exponent $a$ close to 0.2. An exception to this trend is the observation of steady state creep (with $a=1$) in specially-oriented, single $\alpha-\beta$ colony crystals at stress levels below the 0.2% yield strength when tested at room temperature\[12\]. Figure 2.15 also shows that the $A$ value varies with the scale of the microstructure. The basketweave microstructure is found to be more creep resistant than the colony microstructure. The $A$ value also depends upon the stress, with lower creep stresses leading to smaller $A$ values.

This low temperature creep behavior is quite remarkable in terms of its magnitude at a given fraction of the yield strength relative to many other metals and alloys. In the literature one reason proposed to rationalize this unusual creep behavior in titanium alloys is based on the strain rate sensitivity of these alloys \[10, 13\]. However, as we will discuss, the strain rate sensitivity is actually not abnormally high for these materials and is also relatively constant with strain rate and strain. One of the purposes of this analysis is to show that consideration of both the strain rate sensitivity and the strain hardening characteristics can rationalize the low temperature creep behavior of titanium alloys.
5.2 Correlation of Creep and Constant Strain Rate Behavior

Lubhan [14] made one of the earliest attempts to correlate the creep and constant strain rate behavior in metals. He derived a relationship between the material constants, strain hardening exponent, $n$, the strain rate sensitivity, $m$, and the creep deceleration rate $p$ as,

$$p = -\frac{n}{m}$$

where creep rate $\dot{\varepsilon} = C \cdot \varepsilon^p$. However, an explicit relationship for the creep constant $C$, was not derived. Later, Lubhan and Felgar [8] showed examples correlating constant strain rate and creep behavior; but, their approach was to predict the constant strain rate law from a family of creep curves. Chu [15] followed the approach of Lubhan and Felgar to correlate the creep behavior of Ti-6Al-2Nb-1Ta-0.8Mo (Ti-6211) alloy with the constant strain rate behavior. We will now present an extension of this work, and derive a simple analytical result with which one can in principle predict long-time creep behavior from the analysis of short-term, constant strain rate tests.

Let us assume the constitutive behavior of the material can be expressed in two different but consistent forms:

$$\varepsilon = f(t, \sigma) \quad (5.2)$$

and

$$\sigma = h(\varepsilon) \cdot g(\dot{\varepsilon}) \quad (5.3)$$

where, the first and second forms are particularly appropriate for creep and controlled strain rate testing, respectively.
The important assumption in equation 5.3 is that strain is the key state variable such that the flow stress is not a function of prior strain rate history. In terms of creep behavior, this assumption implies that at a given value of strain and instantaneous strain rate, it is not important if the strain was accumulated at a high or low strain rate. This assumption is reasonable for most structural materials at low homologous temperatures, because under these conditions one does not expect the yield strength of the material change with time.

For many metals and alloys the specific form of equation 5.3 is well represented by the strain-rate-sensitive Hollomon flow equation, where the flow stress is a function of both strain and strain rate:

$$\sigma = K \varepsilon^n \dot{\varepsilon}^m$$

(5.4)

where $\sigma$ is the true stress, $\varepsilon$ the true plastic strain and $\dot{\varepsilon}$ is the plastic strain rate. $K$ is the strength parameter, $n$ is the strain hardening exponent, and $m$ is the strain rate sensitivity. In the next section, experimental results will be presented that show that the Hollomon law is a good description of the plastic flow behavior of the two titanium alloys studied in this work, at room temperature.

As discussed above, the creep behavior of titanium alloys can be represented by a power law in time (equation 5.1). In order to achieve the goal of predicting the creep response from the constant strain rate response, one needs to derive a relationship between the creep law constants, $A$ and $a$, and the plastic flow law constants $K$, $n$ and $m$.

From equation 4 the strain rate during plastic flow is given by,
Rearranging equation 5.5, we get,

\[ \dot{\varepsilon} = \frac{d\varepsilon}{dt} = \left(\frac{\sigma}{K}\right)^\frac{1}{m} \cdot \varepsilon^{\frac{n}{m}} \]  

(5.5)

Integrating equation 5.6 for constant stress conditions, and assuming the strength parameter, $K$, to be independent of time, we obtain,

\[
\int \varepsilon^{\frac{n}{m}} d\varepsilon = \int \left(\frac{\sigma}{K}\right)^\frac{1}{m} \cdot dt
\]

(5.6)

Equation 5.7 has the same form as equation 5.1. Comparing equation 5.7 with equation 5.1, one obtains the creep constants $a$ and $A$,

\[ a = \left(\frac{m}{m + n}\right) \]  

(5.8)

\[ A = \left(\frac{\sigma}{K}\right)^\frac{1}{m+n} \cdot \left(\frac{m + n}{m}\right)^\frac{m}{m+n} \]  

(5.9)

Equations 5.8 and 5.9 are the two equations which relate the constant strain rate behavior to the creep behavior. Thus, the Hollomon equation directly implies a power law form for
the time-dependence of creep. It should be noted that as \( n \) tends to zero, \( a \) approaches a value of 1, which would be steady state flow. Therefore, the Hollomon form can intrinsically capture both normal primary creep as well as steady state flow.

One can now measure the material quantities \( K, n \) and \( m \) from constant strain rate experiments. Assuming the form holds, this can be accomplished with as little as one constant strain rate experiment plus a strain-rate jump. Using these measured quantities, we will now show that a reasonable prediction of the long-term creep response (including the stress dependence) for titanium alloys at room temperature can be obtained.

In principle this type of correlation should be valid in the strain range where the strain rate sensitive Hollomon form (equation 5.4) represents the constant strain rate flow behavior. In such cases the power law form of equation 5.1 naturally represents the creep behavior. During the room temperature creep deformation of titanium alloys one accumulates only a few percent strain, typically less than 3%. Hence, in this study plastic deformation has been limited to less than 3% strain in both constant strain rate and creep deformation.

It is important to emphasize at this point that this correlation assumes that the microstructural hardness \( h(\varepsilon) \) of the material depends uniquely on strain. Further, hardness is not affected by strain rate history. These assumptions would appear to be fulfilled in the present case for the following reasons: (a) Figure 5.1 shows the true plastic strain versus true stress of Ti-6Al constant strain rate data. The plot shows that the flow curve of the material after a strain rate jump follows the flow curve of a monotonic test at the higher strain rate. This indicates that there is little strain rate history effect in the
material. (b) In interrupted creep tests, where the sample was unloaded and then reloaded after a 24 hour waiting period, we did not observe significant difference in the creep rates immediately before unloading and after reloading. (c) During this ambient temperature deformation, diffusion-assisted microstructural changes such as subgrain formation are probably not significant. It will be shown later in chapter 6 that this reasoning is borne out by microstructure observations.

5.3 Experimental Correlation of Creep and Constant Strain Rate Behavior

Figures 5.2 and 5.3 are log-log plots of true plastic strain versus true stress for both Ti-6Al and Ti-6242 tested at several strain rates. The data at different strain rates plot as straight lines, indicating that the Hollomon flow equation is a reasonable description of plastic flow in these cases. There is some curvature of the data, which can be seen particularly for the case of Ti-6242 at small strains (<0.003). This curvature cannot be attributed to the fact that the actual plastic strain rate is changing rapidly during the early portion of the test. The curved portions of the plots are for strains beyond those for which the elastic to plastic strain transition has occurred. The flow curves at different strain rates appear as parallel lines with the slope of \( n \). The fact that these curves are parallel for different strain rates indicates that \( n \) does not vary strongly with strain rate, lending support for our use of the Hollomon form. The measured Hollomon parameters for both Ti-6Al and Ti-6242 are tabulated in table 5.1. The values of \( n \) and \( K \) listed in table 5.1 are those determined based on the larger strain data (\( \varepsilon > 0.005 \)).

Using the average of the measured material constants \( K, n \) and \( m \) shown in table 5.1, the creep parameters \( A \) and \( a \) were calculated for both Ti-6Al and Ti-6242. These values are shown in tables 5.2 and 5.3.
Figure 5.4 shows the creep response of Ti-6Al tested at a stress level of 716 MPa and Ti-
6242 tested at a stress level of 931 MPa. This is a linear-linear plot of creep time versus
creep strain. The creep response is primary in nature as in other titanium alloys. The
creep rate is continuously decreasing during the test approaching creep rates of the order
of $10^{-9}$ s$^{-1}$ and steady state creep is not reached in these tests.

Figures 5.5 and 5.6 show the comparison between the predicted and actual creep response
of the two alloys Ti-6Al and Ti-6242 for two different stress levels. The plots show that
the predicted creep response reproduces reasonably well both the time and stress
dependence of the actual creep response. The prediction is especially good for the
commercially-important Ti-6242 alloy. Significant deviation from the linear prediction is
consistently observed at small strains and times. While these discrepancies are
insignificant with respect to the prediction of total accumulated strains at longer times,
they do suggest that the exhaustion creep transient may be comprised of several different
regimes of behavior. The correlation between the creep transient and evolution of slip
traces will be described below which lends some insight into the origin of these different
regimes of creep as a function of time.

From the data shown in the previous section, it appears that one can predict the creep
response of a titanium alloy reasonably well from constant strain rate data. This is clearly
useful considering that the constant strain rate experiments can be accomplished rapidly
compared with a set of creep experiments. Using these data one can then predict the creep
response without performing many creep tests.
5.4 Comparison with Primary Creep in Other Metals and Alloys

Another use of the phenomenological description of creep discussed above is that it represents a convenient framework with which to compare behaviors in different metals. Table 5.4 compares the time exponent $a$ for creep of titanium alloys with published data for other metals tested at various fractions of the homologous temperature. It is obvious that the time exponent is considerably larger for titanium alloys than for Ag and Al, for example, at room temperature. Table 5.4 also lists $a$ values for Ni and Cr-Mo steel tested at significantly higher fractions of their homologous temperatures. These $a$ values are comparable to those observed in titanium alloys at room temperature. These data show that titanium alloys exhibit creep behavior at room temperature which would normally be observed at much higher temperatures in metals such as Ni and Cr-Mo steels.

It was mentioned above that in the literature it was suggested that the room temperature creep of titanium alloys was due to their high strain rate sensitivity[10, 13]. Based on their work on Ti-6Al-4V (Ti-6-4), Odegard and Thompson argue that, during creep, the material is deforming at ever decreasing strain rates, approaching the order of $10^{-8}$ s$^{-1}$ to $10^{-9}$ s$^{-1}$. If the strain rate sensitivity were very large, then the material would effectively become softer as the strain rate decreases so that the applied stress might ultimately approach or exceed the yield strength. However, tables 5.5 and 5.6 show the initial creep rates from the creep tests done on both Ti-6Al and Ti-6242 at different stress levels, which indicates that the initial creep rates are comparable to the strain rates one uses in constant strain rate tests (except for the test at the lower stress level for Ti-6242).

Figure 5.7 shows calculated creep curves for three model materials having different flow properties during constant strain rate deformation. All three materials are assumed to
have a yield strength of 700 MPa at a strain rate of $1 \times 10^{-4} \text{s}^{-1}$. The $K$ value was calculated for the three materials using the strain rate sensitive Hollomon law (equation 5.4). The material constants used for the three model materials A, B and C are listed in figure 5.7. Using the material constants $K$, $n$, $m$ and equations 5.9 and 5.10, the creep power law (equation 5.1) constants $A$ and $a$ were calculated. The three creep curves were calculated using the calculated $A$ and $a$ values and using a creep stress of 630 MPa (0.9YS).

Comparing the creep behavior of material A with B, one can observe that B has accumulated smaller creep strains at long times. Material A has flow properties similar to titanium alloys at room temperature. Both materials A and B have the same $m$ value but material B has a much larger $n$ value than A. From this one might conclude that all materials with a low work hardening exponent might exhibit poor creep properties. But comparison of material B with C shows that eventhough both materials have the same high $n$ value, material C has much less long-term creep resistance because it has a much higher $m$ value.

The $n$ and $m$ values of titanium alloys are compared with other metals in Table 5.7. We see that the $m$ values for titanium alloys are not abnormally high and are, in fact, comparable to both copper, ferritic steels and stainless steels. However, if one considers the strain hardening exponent $n$, its value is abnormally low for titanium alloys as compared to these other metals. The table also shows the $a$ values predicted for different materials based on the preceding analysis (using equation 5.8). This comparison clearly indicates that for titanium alloys, the high $a$ values observed in room temperature creep are due to extremely low $n$ values, while the $m$ values are only moderately large. It is this particular combination that leads to the unusual room temperature creep behavior observed in titanium alloys.
5.5 Summary

Based on the use of the strain-rate-sensitive Hollomon flow law, it has been shown that one can predict reasonably well the low temperature creep response from a limited set of constant strain rate data for materials that do not show a time dependence of hardness. The creep time exponent $\alpha$ is abnormally high for room temperature creep of titanium alloys when compared to a number of metals and alloys. One can rationalize the room temperature creep of titanium alloys as due to abnormally low $n$ and moderate $m$ values observed in these materials.
References


Figure 5.1: True plastic strain vs true stress plot of Ti-6Al constant strain rate data. Plot shows that the flow curve after a strain rate jump follows the monotonic flow curve at the higher strain rate. This indicates little strain rate history effect in the material.

Figure 5.2: Log true plastic strain versus log true stress plot of Ti-6Al constant strain rate data. The plot also shows the strain-rate-sensitive Hollomon law fit for the data.
Figure 5.3: Log true plastic strain versus log true stress plot of Ti-6242 constant strain rate data. The plot also shows the strain-rate-sensitive Hollomon law fit for the large strain data.

Figure 5.4: Linear-linear plot of creep time versus creep strain of Ti-6Al and Ti-6242 tested at 716 MPa and 931 MPa respectively. The plot shows that creep response is primary in nature with the creep rate continuously decreasing with time.
Figure 5.5: Comparison of predicted creep response and experimental creep response for Ti-6Al. Creep curves for two stress levels corresponding to 0.94 and 0.80 of the 0.2% yield strength measured at a strain rate of $1.5 \times 10^{-4} \text{ s}^{-1}$ are plotted. The predicted curve is calculated using the Hollomon parameters tabulated in Table 5.1.

Figure 5.6: Comparison of predicted creep response and experimental creep response for Ti-6242. Creep curves for two stress levels corresponding to 0.95 and 0.84 of the 0.2% yield strength measured at a strain rate of $1.34 \times 10^{-4} \text{ s}^{-1}$ are plotted. The predicted curve is calculated using the Hollomon parameters tabulated in Table 5.1.
Figure 5.7: Comparison of calculated creep response of three model materials A, B and C with different material flow properties. The creep stress for all the materials are 630 MPa (0.9YS). See text for details.
<table>
<thead>
<tr>
<th>Material</th>
<th>Strain Rate (s⁻¹)</th>
<th>K (MPa)</th>
<th>n</th>
<th>m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al</td>
<td>8.4x10⁻¹</td>
<td>1161</td>
<td>0.042</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.5x10⁻⁴</td>
<td>1136</td>
<td>0.039</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.68x10⁻⁵</td>
<td>1133</td>
<td>0.039</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.5x10⁻⁴ to 1.3x10⁻³</td>
<td></td>
<td>0.019</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.68x10⁻⁵ to 1.72x10⁻⁴</td>
<td></td>
<td>0.018</td>
<td></td>
</tr>
<tr>
<td>Average Parameters</td>
<td></td>
<td>1143</td>
<td>0.04</td>
<td>0.0185</td>
</tr>
<tr>
<td>Ti-6242</td>
<td>1.34x10⁻¹</td>
<td>1425</td>
<td>0.048</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.44x10⁻⁵</td>
<td>1435</td>
<td>0.052</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.34x10⁻⁴ to 1.42x10⁻³</td>
<td></td>
<td>0.01</td>
<td></td>
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<tr>
<td></td>
<td>1.44x10⁻⁵ to 1.43x10⁻⁴</td>
<td></td>
<td>0.01</td>
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</tr>
<tr>
<td>Average Parameters</td>
<td></td>
<td>1430</td>
<td>0.05</td>
<td>0.01</td>
</tr>
</tbody>
</table>

Table 5.1: Measured Hollomon parameters for Ti-6Al and Ti-6242.

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>A</th>
<th>a</th>
</tr>
</thead>
<tbody>
<tr>
<td>827.5</td>
<td>0.000148</td>
<td>0.17</td>
</tr>
<tr>
<td>931</td>
<td>0.0011</td>
<td>0.17</td>
</tr>
</tbody>
</table>

Table 5.2: Calculated creep parameters for Ti-6242.
Table 5.3: Calculated creep parameters for Ti-6Al.

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>A</th>
<th>a</th>
</tr>
</thead>
<tbody>
<tr>
<td>606</td>
<td>0.000030</td>
<td>0.32</td>
</tr>
<tr>
<td>716</td>
<td>0.00053</td>
<td>0.32</td>
</tr>
</tbody>
</table>

Table 5.4: Comparison of time exponent $a$ in creep of various conventional materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Temp ($T/T_m$)</th>
<th>a</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>0.15</td>
<td>0.21</td>
<td>[9]</td>
</tr>
<tr>
<td>Al-61ST</td>
<td>0.3</td>
<td>0.03</td>
<td>[8]</td>
</tr>
<tr>
<td>Pure Ag</td>
<td>0.24</td>
<td>0.03</td>
<td>[8]</td>
</tr>
<tr>
<td>Pure Ni</td>
<td>0.62</td>
<td>0.21</td>
<td>[16]</td>
</tr>
<tr>
<td>Cr-Mo Steel</td>
<td>0.4</td>
<td>0.24</td>
<td>[8]</td>
</tr>
</tbody>
</table>

Table 5.5: Initial creep rates measured in Ti-6Al.

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>Initial Creep Rates ($s^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>606</td>
<td>$1.7 \times 10^{-5}$</td>
</tr>
<tr>
<td>716</td>
<td>$9.14 \times 10^{-5}$</td>
</tr>
</tbody>
</table>
### Table 5.6: Initial creep rates measured in Ti-6242.

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>Initial Creep Rate ($s^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>827.5</td>
<td>$3.625 \times 10^{-7}$</td>
</tr>
<tr>
<td>931</td>
<td>$8.4 \times 10^{-5}$</td>
</tr>
</tbody>
</table>

### Table 5.7: Comparison of $n$ and $m$ for conventional alloys with titanium alloys.

<table>
<thead>
<tr>
<th>Material</th>
<th>$m$</th>
<th>$n$</th>
<th>$a = m / (n + m)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti Alloys</td>
<td>0.015</td>
<td>0.03-0.07</td>
<td>0.3-0.18</td>
</tr>
<tr>
<td>OFHC Cu</td>
<td>0.01[17]</td>
<td>0.4[18]</td>
<td>0.02</td>
</tr>
<tr>
<td>304 SS</td>
<td>0.02[17]</td>
<td>0.5[18]</td>
<td>0.04</td>
</tr>
<tr>
<td>Ferritic Steels</td>
<td>0.02[19]</td>
<td>0.1-0.3[19, 20]</td>
<td>0.16-0.06</td>
</tr>
<tr>
<td>Al Alloys</td>
<td>$&lt;0.005[21]$</td>
<td>0.2-0.3[22, 23]</td>
<td>0.02-0.0</td>
</tr>
</tbody>
</table>
CHAPTER 6

EFFECT OF SHORT-RANGE ORDER (SRO) ON THE PRIMARY CREEP BEHAVIOR OF TITANIUM ALLOYS

6.1 Introduction

In the literature, short-range order (SRO) of Ti and Al atoms was presumed to occur in dilute Ti-Al alloys[1-4]. This was thought to influence the deformation behavior in the α phase of titanium alloys[1, 4, 5]. In chapter 4 neutron diffraction data was presented for a Ti-6Al alloy after various heat treatments to modify the SRO state, which provided direct diffraction evidence for the presence of SRO in Ti-Al alloys for the first time. Phenomenological analysis of room temperature creep presented in chapter 5 indicated that the most significant factor that influences the room temperature creep of titanium alloys is their low work-hardening exponents. In this chapter, effect of SRO on the room temperature creep behavior of a Ti-6Al alloy will be presented. The deformation behavior was characterized using both, optical and transmission electron microscopy (TEM). Deformation behavior in the α phase of many commercial α, near-α and α/β titanium alloys is similar to that observed in Ti-6Al. Based on the microstructural studies, a possible explanation for the observed low work-hardening exponents in titanium alloys is provided. A dislocation model will be proposed to explain the deformation behavior
observed after the various heat treatments to modify the SRO state. In light of which the room temperature creep behavior of other titanium alloys will be discussed.

6.2 Effect of SRO on room temperature creep of Ti-6Al

Two kinds of heat treatments were done to modify the SRO state. All samples were annealed at 900°C for 24h. After the high temperature anneal, samples were cooled at different rates to room temperature and were tested in the as cooled condition. Samples were also subsequently aged at various temperatures for different times to modify the SRO state. The heat treatments and labels of the samples are listed in table 3.3. Creep tests were performed at the same stress level of 552 MPa on all samples.

Figure 6.1 shows the creep response of five different samples that were ice water quenched (IWQ) to room temperature after the high temperature anneal. This is a log-log plot of creep time versus creep strain. The initial creep rates, creep strain at t=1s, creep rate at t=950s are listed in table 6.1. In the literature, room temperature creep behavior of titanium alloys have been represented by a power law in time (equation 5.1). Consistent deviation from the power law behavior has been observed at early times as discussed in chapter 5. In the case of IWQ samples there are three regimes with different power law time exponents. The initial regime has a lower time exponent \( a_i \), i.e. higher exhaustion rate, a second regime where the time exponent is high \( a_{ii} \), i.e. lower exhaustion rate and there is a third, long time regime with a smaller time exponent again \( a_{iii} \), i.e. higher exhaustion rate. This is illustrated in figure 6.10, which is a log-log of plot of creep strain versus creep rate. The time exponents are listed in table 6.1. All five samples exhibit similar primary transients and the instantaneous creep strains for all the samples are very similar. Initial creep rates \( \approx 10^4 \text{ s}^{-1} \) and creep strains \( \approx 10^{-4} \) are relatively high for all five
samples. But, there is a significant spread in the amount of total creep strains between the different samples. IWQ-1 sample is the most creep resistant and IWQ-2 is the weakest. In the case of IWQ-2 sample the exhaustion rates in regimes I and II are much lower than the other samples, this is indicated by the larger creep rate at t=950s. Hence it accumulates the largest creep strains. The long time creep exponent is very similar in all the cases. Finally, the transition to the long time behavior seems occurs at longer times, when the strain accumulation in regimes I and II is lower. Also, the creep strain range at which the transition to long time regime appears to occur within a strain range of 0.003-0.005.

Namboodhiri et al[2] studied the Ti-rich end of the Ti-Al phase diagram. They studied the change in resistivity after various heat treatments in alloys with aluminum content ranging from 3.94wt% to 11.77 wt%. For a Ti-5.9wt%Al alloy (with an O₂ content of 430wtppm), they observed a peak in resistivity after ageing at 505°C for 6 hours. Whereas, they observed faint superlattice reflections corresponding to the α₂ (Ti₃Al) phase in the same alloy only after ageing for 400h at 505°C. Based on these observations and other evidence, they concluded that the peak in resistivity after an age of 6h at 505°C is due to a peak in SRO state of Ti-Al atoms.

Figure 6.2 shows the creep response after ageing IWQ samples to various times at 500°C. These samples also exhibit three regimes of creep exhaustion rates as indicated by the shape of the primary transient in log-log plots of creep time versus creep strain. The initial creep rate, creep strain at t=1s and creep rate at t=950s along with the three α values are listed in table 6.2. Initial creep strains are very similar after the different treatments, but the initial creep rate seems to increase with ageing time with the
exception of the IWQ-500C-10h sample. At long times, creep resistance of the IWQ-500C-50h sample was the weakest and the IWQ-500C-5h was the most creep resistant. But the differences in the creep resistance between IWQ-500C-0.5h, IWQ-500C-5h and IWQ-500C-10h samples are relatively small. There are some subtle differences in the primary transients of the different samples. The sample that was aged for 10h showed a lower exhaustion rate in both regimes I and II, as indicated by the relatively high values of $a_1$ and $a_{ii}$. Hence, accumulated creep strains similar to the IWQ-500C-0.5h and IWQ500C-5h samples at long times even though the initial strains and strain rates were a little lower. The IWQ-500C-5h sample showed the highest exhaustion rate in regime II and was the most creep resistant. In the case of IWQ-500C-0.5h sample, it showed very low time exponent indicating rapid exhaustion in regime I. But, the creep exhaustion rate in regime II was the lowest (high $a_{ii}$) and hence, at long times it accumulated creep strains similar to other samples. The IWQ-500C-50h sample exhibited intermediate creep exhaustion rates in both regimes I and II, but the initial creep strains and rates were high and accumulated the largest creep strains at long times. Creep behavior after ageing for different times does not appear to exhibit classic age hardening type behavior that one might expect based on the results of Namboodhiri et al, and the differences in creep behavior are very subtle.

Additional ageing experiments were conducted at different temperatures after the IWQ treatment. Samples were aged at 350°C for 100h and at 600°C for 1h. These data are compared with the creep response of the IWQ-500C-5h in figure 6.3. Namboodhiri et al[2] and Shull et al[6] have reported that the $\alpha/\alpha_2+\alpha$ transus for a Ti-6Al alloy is $\approx$550°C. Hence, the IWQ-600C-1h sample was aged above the two phase field. Creep transient of this sample is similar to the IWQ-500C-5h and showed three regimes of
exhaustion. The initial creep strain and strain rate of this sample is similar to those observed in the samples aged at 500°C. The creep time exponent of regime II (a_t) is only marginally higher than that observed in the samples aged at 500°C, but the overall creep resistance is lower than the IWQ-500C-5h sample. Because, this regime of lower exhaustion rate occurs for a longer time and the sample accumulates larger creep strains.

Figure 6.3 also shows the creep response of the sample aged at 350°C for 100h. Long time creep strains are similar to the sample aged at 500°C for 5h, but the 350°C sample is a little stronger. The initial creep strain and creep rate are one half of that of the IWQ-500C-5h sample. Even though the $a$ value of the initial regime is higher than the IWQ-500C-5h, this occurs for a longer time and regime II of lower exhaustion occurs for lesser time. All these factors cause the sample to accumulate lower creep strain after long times.

Several samples were air cooled (AC) to room temperature following the high temperature anneal at 900°C for 24h. Creep response of these samples are shown in figure 6.4. Initial creep rates, creep strains and other creep data are shown in table 6.3. Long time creep behavior of all AC samples are very similar. Spread in the total creep strains between the different samples is small compared to the IWQ samples. Samples AC-1 and AC-2 have identical long time creep transient, but the initial creep transients are a little different. These samples were annealed in the same batch. Whereas, samples AC-3 and AC-4 were annealed in two different batches, even in this case long time creep transients are identical. This set of two samples are more creep resistant at longer times. The initial creep rates and creep strains of AC samples are lower than that observed in the IWQ samples or the IWQ-age samples. But, if one compares the creep rate at $t=950$s, the creep rates of the AC samples and the IWQ/IWQ-age samples are very similar. This is
because, the creep transient of these samples are very different from both the IWQ sample and the IWQ-age samples. In the case of AC samples the primary transient essentially has only two regimes of different exhaustion rates. An initial regime of very low exhaustion rate (high $a$ values) and a final long time regime of higher exhaustion (low $a$ values). In the case of samples AC-1 and AC-3, there is a regime that looks like an initial regime of rapid exhaustion. This is actually a regime of very low creep rates ($\approx 10^{-9}$ to $10^{-8} \text{s}^{-1}$) for tens of seconds, after which there is a very short inverse primary regime with increasing creep rates for a few seconds. This is followed by a regime of normal primary transient. If one compares the time exponent $a_{\text{II}}$ of the AC samples with the time exponent $a_{\text{II}}$ of the IWQ and IWQ-age samples, it is obvious that the time exponent of AC samples are much higher. The regime of very low exhaustion occurs for a long time in AC samples, hence, they accumulate significant creep strains at long times, even though they are much stronger at short times. One sample, AC-2 does show three regimes of creep exhaustion, but the $a_{\text{I}}$ value is very high.

Figure 6.5 compares the creep response of an AC sample (AC-1) and samples that were aged after the AC treatment. Samples were aged at 350°C for 5h and at 400°C for 1h and 5h. The initial creep strain, creep rates and other creep parameters are listed in table 6.3 for comparison. Long time creep transient of all samples are very similar. The alloy becomes more creep resistant with ageing because the AC-400C-5h sample is the strongest, followed by AC-400C-1h and AC-350C-5h samples. But, the differences in the total creep strain levels between the aged samples and the as AC sample is not very large. The early time creep transients of the different AC-age samples are very different. The initial creep strain is larger than that observed in the AC samples and the initial creep rates are also higher. The $a_{\text{II}}$ value is high, but comparable to the AC samples. Creep time
exponent $a_{m}$ is lower than that observed in AC samples. In the case of AC-350C-5h sample, three regimes of different exhaustion rates were observed. The $a$ values corresponding to these regimes are listed in table 6.3. Sample AC-400C-1h exhibited an initial regime of very low creep rates ($\approx 10^{-9}$ to $10^{-4}$ s$^{-1}$) as was observed in AC-1 and AC-3 samples. In this case the initial creep strains were higher than that in AC-1 and AC-3 samples. After the initial regime there was an inverse primary, where the creep rate increased for a few seconds, this was followed by a normal primary. The normal primary had three regimes of exhaustion, the $a$ values corresponding to these are listed in table 6.3. Regime II has a time exponent of 0.96, indicating close to steady state behavior for a short time. The final exponent is similar to what was observed in AC samples. AC-400C-5h sample, also exhibited three regimes of normal primary. The initial creep strains and the creep rates were very high compared to the AC samples. In this sample the exhaustion rate is very high in regime I ($a_i=0.08$) and this regime occurs for longer times. Exhaustion rate in regime II is comparable to other AC/AC-age samples, but this regime of lower exhaustion occurs for only shorter times in this sample. Hence, this sample showed lower creep strain after long times.

Samples were also aged above the $\alpha/\alpha_2+\alpha$ transus at 650$^\circ$C for 24h after the high temperature anneal at 900$^\circ$C for 24h. Two different types of heat treatments were done. In one case the sample was IWQ to room temperature and then aged at 650$^\circ$C. Samples were directly step quenched to 650$^\circ$C in the second case. Creep response of the samples after these two treatments are compared in figure 6.6. There is no significant difference in the creep response between the different samples. The creep parameters are listed in table 6.4. Initial creep rates and strains are identical for all three samples. Two samples, IWQ-650C-24h and SQ-650C-24h-1 showed three regimes in the primary transient, whereas
sample SQ-650C-24h-2 showed only two regimes in the transient. Further, in the case of SQ-650C-24h-2 sample, the exhaustion rate in regime I was very low and comparable to the rate in regime II. Creep time exponent in regime II is relatively high ($\alpha_{II}=0.6$) for all three samples and this regime occurs for relatively long times. Hence, they accumulate significant creep strains after long times.

Three different step quench (SQ) treatments were done to get a stronger more uniform SRO state. In these SQ treatments, the samples were quenched to the ageing temperatures after the high temperature anneal at 900°C. One sample was aged at 600°C, which is above the $\alpha/\alpha_2+\alpha$ transus, for 24h. Another sample was aged at 400°C for 5h, which is inside the two phase region. A third sample was step quenched to 600°C and aged for 24h. After this ageing treatment, the sample was ice water quenched to room temperature and then was aged again at 400°C for 5h. Creep response after these three treatments are compared in figure 6.7. There are significant differences in the creep transient of the three samples. Creep parameters for the three sample are listed in table 6.4. All three samples exhibited a regime of very low initial creep rates similar to the sample, AC-1. In the case of SQ-600C-24h sample the initial creep strains were much higher than the other two samples. After this initial low creep rate regime, in this sample, there was an inverse primary lasting a few seconds and then a normal primary transient. There were only two regimes in the normal primary transient of this sample. Both SQ-400C-5h and SQ-600C-24h-400C-5h samples exhibited very different primary transients compared to all the samples discussed above. Initial creep rates and creep strains were very low after loading. After this “incubation” period there was an inverse primary regime, for few seconds. This was followed by a normal primary for hundreds of seconds. After this first normal primary, there was a second inverse primary, where the creep rate increased for
thousands of seconds. This was followed by another normal primary regime. In other words there were two inverse primary regimes in these two samples. One inverse primary lasted for a few seconds after the initial "incubation period" and then a second regime where it lasted for thousands of seconds after a first normal primary. The "incubation" regime lasted for \( \approx 10 \) s in the case of SQ-400C-5h. The creep exhaustion rate in the first primary after this was very low \((a=0.87)\). Comparing this with the SQ-600C-24h-400C-5h sample, in which, the "incubation" time was \( \approx 40 \) s. The exhaustion rate in the first normal primary regime was much higher \((a=0.48)\). The exhaustion rate during the second normal primary regime was comparable in both samples \((a\approx0.32-0.35)\). Creep strains after long times were comparable between the SQ-600C-24h sample and the SQ-400C-5h sample and the latter was a little higher. The SQ-600C-24h-400C-5h sample was significantly stronger than both the samples. The differences between the primary transients of SQ-400C-5h sample and SQ-600C-24h-400C-5h samples are illustrated in figure 6.9.

Figure 6.8 compares the creep response of samples after different heat treatments to alter the SRO state. This plot summarizes the various creep transients that were observed due to the different treatments. The long time creep response is very similar for all the samples. Two samples were much stronger than all the rest of the samples. These were IWQ-1 and the SQ-600C-24h-400C-5h samples. But the primary transient is very different between these two samples. The SQ-600C-24h-400C-5h sample is much more creep resistant at short times, but it shows two regimes of inverse primary and hence accumulates creep strains similar to IWQ-1 sample at long times. Both these samples accumulate creep strain about 1.6 times lower than all the other samples. Because the
long time exponent are similar for all the samples, these two samples will have accumulated lower strains even after a very long time.

To summarize, creep tests after different heat treatments show significant differences in the primary transient. As first discussed in chapter 5, there are consistent deviations from the power law behavior at early times. Dramatic differences in the transient occur at early times, whereas the long time behavior is more or less similar between the different samples. In the case of IWQ samples there are significant differences in the total creep strains between different samples. The observed primary transients among the different heat treatments can be classified into three kinds of primary transients. The IWQ and IWQ-age samples exhibit a primary transient with three regimes of different exhaustion rates. The AC samples showed only two regimes of different exhaustion rates in the primary transient. Initial creep rates of two air cooled samples AC-1 and AC-3 were very low \(10^{-9}-10^{-4} \text{ s}^{-1}\). Such low initial creep rates ("incubation" period) was also observed in the AC-400C-1h, SQ-600C-24h, SQ-400C-5h and SQ-600C-24h-400C-5h samples. Although, several samples showed an initial regime of very low creep rates, the instantaneous strain was found to be significantly different between these samples. Both SQ-400C-5h and SQ-600C-24h-400C-5h samples showed very different primary transients compared to the other samples. There was an initial regime of very low creep rates, was followed by a regime of inverse and normal primary in that sequence. This is similar to the AC samples. In addition these two samples showed a second inverse primary regime which occurred over several thousand seconds and this was followed by another normal primary regime. Finally, the SQ-600C-24h-400C-5h sample was found to be more creep resistant than both SQ-400C-5h and the SQ-600C-24h samples. The three different types of transients that were observed are illustrated in figure 6.10.
6.3 Characterization of deformation microstructure

As discussed above, there are significant differences in the primary transients after different heat treatments. Both optical and transmission electron microscopy was used to study the microstructural characteristics of the deformation. These studies provided insight into dislocation level processes as well as insight into the form of the creep transient.

6.3.1 Operative slip systems during room temperature creep

TEM studies were conducted to study the dislocation structures as well as to determine the operative slip systems after creep deformation at room temperature. Dislocation burgers vector was determined by g.b invisibility analysis and the slip plane was determined from slip trace analysis. Determination of burgers vector and slip plane is illustrated in figure 6.11 in an IWQ-350C-100h sample. In this grain only two slip systems were active. The primary slip system was a prism system (\(\bar{T}100\)) [\(1\overline{1}20\)] and the secondary system was a basal slip system (0001) [\(\bar{T}2\bar{T}0\)]. In a few cases, the slip plane could not be determined unambiguously from tilting experiments. For these, slip trace analysis was conducted using the program Desktop Microscopist 2.0 to determine the slip plane. The foil normal was also determined for many of the samples analyzed. This was also done using Desktop Microscopist 2.0. The zone axis that was within 1 to 2 degrees from the actual foil normal was designated as the foil normal for the grain. The operative slip systems in many of the samples that were analyzed is presented in tables 6.5-6.7. Ranking of the operative slip systems within a grain and schmid factors for the operative slip systems are also shown in tables 6.5-6.7. The slip system ranking is only a qualitative
ranking based on the amount of activity of a particular slip system in the analyzed grain. One can observe from the tables that prism \(<a>\) slip was the most frequently observed primary slip system. Basal \(<a>\) slip was also observed frequently and was the most common secondary slip system. Pyramidal \(<a>\) slip was active in a few cases. In these tables, only data on \(<a>\) type slip systems were presented. It should be noted that \(<c+a>\) pyramidal slip was frequently observed in many of the grains. Detailed analyses of \(<c+a>\) dislocations was not done. Even though the creep samples were deformed to less than 1% plastic strain in most cases, \(<c+a>\) dislocation activity was observed and primarily near grain boundaries. An example of this is shown in figure 6.12, which shows a bright field image of \(<c+a>\) dislocations near a grain boundary in an AC sample. Presumably \(<c+a>\) dislocations were activated to provide strain continuity across neighboring grains, hence they are present mainly near grain boundaries. These \(<c+a>\) dislocations were also observed to move into the grain interiors in many cases (see figure 6.13b). There were cases where extensive \(<c+a>\) slip occurred inside the grains.

6.3.2 Observed dislocation structures after creep deformation

Deformation microstructures after creep, of samples with different SRO state, were characterized using TEM. Figure 6.13 shows the deformation microstructure of an air cooled sample (AC-1). In this grain the observed slip system is \(\{100\} [11\bar{2}0]\) and there is \(<c+a>\) dislocation activity as well. The deformation is extremely planar. All the deformation is concentrated in long dislocation pile-ups and there is little dislocation activity outside of these bands. Extrapolated location of the dislocation source (S) is shown in 6.13b. That the dislocations are of opposite signs on either side of the source location can be determined by the change in dislocation lobe contrast on either side. Detailed discussion about the dislocation sources in the Ti-6Al alloy will be made in 125
section 6.3.3. Planar deformation was observed in many grains in the AC sample, but the deformation in some grains was observed to be less planar than what is shown in figure 6.13. Deformation microstructure of a SQ-600C-24h-400C-5h sample is presented in figure 6.14. It shows that deformation is extremely planar in this sample as well. In general, the deformation was observed to be planar in all the samples except IWQ samples, which will be discussed later. It should be reiterated that planar dislocation arrays were observed on prism, basal and pyramidal planes. The degree of planarity is very difficult to quantify, but qualitatively it was observed to decrease as the temperature of ageing was increased to 600°C and above.

Tip of a dislocation array in a SQ-600C-24h-400C-5h sample is shown in figure 6.15. One can observe pairing of the lead four dislocations in the pile-up. The spacing of the dislocations become more or less uniform after the first few dislocations. In this sample pairing of at least the first four dislocations and sometimes the first six dislocations were observed. In all other samples, after various heat treatments, strong pairing of only the first two dislocations was observed.

In the literature, similar dislocation structures have been reported after constant strain rate deformation, in single phase and in the α phase of two phase titanium alloys[1, 4, 7-13]. Deformation propagation through planar dislocation arrays have been reported in several fcc based alloys, Cu-Zn[14], Cu-Al[15], Cu-Mn[16], Ni-Cr[17] and in the disordered γ phase of Ni-based super alloys[18, 19]. Using either x-ray or neutron diffraction techniques it was shown that there is SRO of solute atoms in all of these fcc alloy systems. During deformation the lead dislocation destroys the order in the material. Because it is only short-range order, the trailing dislocations cannot restore the order
completely. This provides significant resistance to the motion of the lead dislocation and lesser resistance for the motion of the trailing dislocations. Self stresses of the trailing dislocations are thought to assist the lead dislocation in overcoming the friction due to destruction of SRO[20]. See section 2.5.1 for a detailed discussion. In single phase titanium rich Ti-Al alloys, slip mode changes from homogeneous to planar deformation with increase in Al content around 4wt% Al[4, 7, 8]. This change in slip mode was thought to occur due to SRO of Ti-Al atoms because there was no direct observation of ordered Ti$_3$Al particles[1, 4, 7]. This suggestion was supported indirectly by the resistivity studies of Namboodhiri et al[2] as discussed before. Neutron diffraction results presented in chapter 4, provided for the first time, direct evidence for the presence of SRO in a Ti-6Al alloy. Further, observation of pairing of lead dislocations has been reported in fcc based alloys with SRO(FCC-SRO)[15, 18, 19]. Cohen and Fine[21, 22] have shown that in fcc alloys with SRO, pairing of lead two dislocations will occur because of partial restoration of SRO by the second dislocation. Based on the neutron diffraction results and observation of pairing of lead dislocations, it is concluded that destruction of SRO causes the planar deformation observed in samples that were air cooled and those aged at lower temperatures to promote SRO in the Ti-6Al alloy. This is also true in general for titanium alloys, because conventional heat treatments will produce a short-range ordered state in the α phase.

One of the remarkable features of the planar dislocation arrays observed in Ti-6Al and in other titanium alloys is that the dislocations in the array are predominantly in screw orientation. This is a unique feature of titanium alloys. There is no such preference for a particular line orientation in FCC-SRO alloys, where dislocations have been observed to be in mixed orientation, in general. Several workers[23-30] have performed atomistic
studies on the dislocation core structure of \(<a>\) type dislocations in \(\alpha\)-Ti. The most recent of these studies was by Girshick et al\([30]\) and they showed that the core structure of \(<a>\) type screw dislocation is spread on both prism and basal planes. Thus the non-planar core of the screw dislocation has to become compact on the slip plane before gliding, during deformation. This makes the screw orientation the slowest moving line orientation. When a dislocation loop is expanding, this would imply that the shape of the loop will become rectilinear with long lengths of screw segments constituting the majority of the line length. During in-situ deformation in a high voltage electron microscope (HVEM), we observed that the edge/ mixed segments ran out of the foil rapidly leaving only a straight screw segments in the foil. Farenc et al\([31]\) based on their in-situ deformation studies of \(\alpha\)-Ti, noted that the mobility of mixed and edge segments of \(<a>\) type dislocation is atleast 50 times higher than that of a screw segment. All these results are consistent with our post-mortem observations of the dislocation microstructure consisting mainly of screw dislocations. Another important implication of the above is that the glide plane softening effect of SRO is strong enough to stabilize arrays of screw dislocations in titanium alloys.

Figure 6.16 shows the deformation microstructure of a sample that was IWQ to room temperature (IWQ-3). The main slip system that was active in this particular grain was \((0001) [T\{2\}0]\). This slip system had the highest schmid factor among the different \(<a>\) type slip systems and more than twice the schmid factor for the three prism slip systems. Therefore, the choice of basal slip as the primary slip system seems to be due to the particular orientation of the grain with respect to the stress axis. The most striking feature of the deformation microstructure is that slip is very homogeneous. In this particular view, one of the grain boundaries (not shown) is on the right side of the figure.
Deformation appears to be planar on the left side, in the grain interior far away from the grain boundary. But one can observe that the slip bands are wavy indicating significant cross slip. Slip becomes diffuse as the deformation proceeds to the right towards the grain boundary. This is a characteristic feature of IWQ samples. Such lateral spread of slip from the initial slip planes due to extensive cross slip was observed during in-situ deformation of IWQ sample when deformation proceeded towards a grain boundary. This is shown in figure 6.17. It has to be reiterated that slip in general, including prism slip, is diffuse in IWQ samples. Neutron diffraction data presented in chapter 4 showed that the diffuse intensity indicating presence of SRO was the weakest for the IWQ sample. We have shown that dislocation pile-ups are the characteristic feature of deformation microstructure of titanium alloys. Since SRO is the weakest in IWQ samples, pile-ups are not stabilized by the destruction of SRO. In other words, cross-slip of screw dislocations are not inhibited in these samples causing very diffuse slip.

6.3.3 Dislocation sources and their operation

In order to understand the observed deformation microstructure and the dislocation level processes causing the different primary transients after various heat treatments, one needs to understand the dislocation sources and their operation in the Ti-6Al alloy. Figure 6.18 shows a montage of few grains in an undeformed IWQ sample. There is only one grown-in dislocation present in the grains. No obvious dislocation sources are present inside the grains. This suggests that the dislocation sources have to be present at the grain boundaries in this alloy, at least initially. It is now well established that grain boundaries can act as dislocation sources (see[32], for a review). Such a grain boundary dislocation source operating in a SQ-600C-24h-400C-5h sample is shown in figure 6.19. Further, during in-situ deformation in a HVEM, it was observed that there were no dislocation
sources operating inside the grains initially. Dislocations were observed to be generated from the edge of the foil, crack-tips and grain boundaries. One such grain boundary source operating during in-situ deformation in a AC sample is shown in figure 6.20. It should be noted that in the in-situ sample the source is oriented to produce dislocations in the screw orientation. This is not the orientation observed to occur in bulk samples as can be seen in figure 6.19. Another interesting feature to note from figure 6.19 is that the dislocations are elongated in the screw orientation even at the source. This is due to the low mobility of screw segments, as discussed in section 6.3.2. One important implication of this is that the dynamics of source operation will be determined by the dynamics of the lateral motion of the screw segments away from the source. This will be discussed further in section 6.5.

6.3.4 Microstructural rationale for observed low n values in titanium alloys

In chapter 5, the phenomenological analysis of room temperature creep indicated that the single most important factor that causes extensive room temperature creep in titanium alloys is their low work hardening exponents. Neutron diffraction studies of Ti-6Al in this work clearly showed the presence of SRO after various heat treatments except in the case of IWQ samples where it was very weak. It was discussed in detail in section 6.3.2 that the presence of SRO causes deformation to be extremely planar in this alloy. During deformation, all the activity is concentrated in slip bands with hundreds of dislocations in them and little dislocation activity outside of these slip bands. Such planar deformation structures have been reported in FCC-SRO alloys as discussed in sections 6.3.2 and 2.5.

Table 6.8 shows the work-hardening rate during single glide or stage I glide at room temperature in single crystalline Cu-Mn[16] alloys as a function of Mn content and in
single crystalline Ni-Cr alloys[33] as a function of Cr content. In the Cu-Mn system there is no significant difference in the work-hardening rates between 1.05 at% Mn and 3.3 at% Mn, but the work hardening rate decreases for the 11 at% Mn alloy. Deformation was observed to be planar in the Cu-11 at% Mn alloy and this was thought to be caused by the presence of SRO. Akhtar and Teghtsoonian [33] studied the plastic deformation behavior of single-crystalline Ni-Cr alloys upto 21.5 at% Cr. The work-hardening rate was observed to decrease with increasing Cr content during stage I deformation. The Ni-20 at% Cr alloy exhibited planar glide[34] and this was attributed to the presence of SRO. These results indicate that the work-hardening rate does change with SRO and appears to decrease with the occurrence of planar glide due to the presence of SRO. It should be emphasized that the work-hardening rates are positive and significant during single slip in these FCC-SRO systems. Comparing this with the response of a single crystalline Ti-5.2 at% Al alloy from the work of Sakai and Fine[5], the Ti-Al alloy exhibited work softening during prismatic single glide.

Table 6.9 shows the work-hardening exponent of a polycrystalline Cu-Al alloy as a function of Al content taken from the work of Kügler et al[35]. The true stress versus true strain data were fit to a Hollomon power law form between a strain level of 0.003 to 0.03 to get the work-hardening exponents. The Cu-10 at% Al and Cu-15 at% Al alloys exhibit planar slip due to the presence of SRO, whereas the Cu-5 at% Al alloy exhibits diffuse slip. There is a trend of decreasing work-hardening exponents when the deformation becomes planar due to SRO even in polycrystals. When one compares the work-hardening exponent of titanium alloys (see table 5.1) with the FCC-SRO system, it is very clear that the work hardening rates in the Cu-Al alloy is much higher than what is observed in titanium alloys. Eventhough planar deformation, promoted due to SRO, tend
to decrease the work-hardening rates, those observed in titanium alloys are very low compared to other systems.

Recently, in the case of austenitic steels (Fe-Ni-Cr\[36-38\] and Fe-Mn-Cr\[38, 39\] steels) it has been shown that addition of nitrogen improves the strength and work-hardening rates significantly. Nitrogen is present as a solid solution strengthener and not in the form of nitrides. Addition of nitrogen promotes planar slip in these alloys\[36-38\]. This was thought to be due to presence of SRO of Fe-N atoms\[40\]. In addition, it has been noted that nitrogen also promotes preference for screw orientation in the deformation microstructures\[36, 37\]. It was shown by Grujicic and Zhou\[41\] that addition of nitrogen alters the core structure of screw dislocations to a non-planar configuration and therefore they are the slowest moving line direction in these steels, similar to titanium alloys. In summary, the deformation microstructure in austenitic steels with nitrogen consists of pile-ups of screw dislocations. This is very similar to titanium alloys, but, addition of nitrogen causes the work-hardening rates to improve in these steels and they are much higher than what is observed in titanium alloys.

An important feature of the deformation microstructure in FCC-SRO alloys is the formation of multipoles (see \[14\]). Multipole formation is observed in the case of high nitrogen austenitic steels as well\[38, 42\]. Dislocation multipoles are configurations, where dislocation arrays of opposite signs on parallel slip planes are locked into low energy configurations. Such structures have been observed in single crystals deformed in single glide as well as in polycrystals in FCC-SRO alloys. Multipole formation is a mechanism for dislocation storage in the material and could provide significant work hardening during deformation, as mobile dislocation densities are reduced due to the
formation of such multipolar configurations. This is consistent with the positive work hardening exhibited by these alloys even during single glide in single crystals [16, 33, 43]. Based on their in-situ deformation studies on polycrystalline Ni-30at%Cr alloy with SRO, Clément et al [17] showed that multipoles do not provide much resistance to the propagation of other dislocation pile-ups. This is reasonable because the multipolar structures will have only short-range interactions with other dislocations. Further, Clément et al studied the obstacles to the propagation of such planar dislocation groups. Hard grain boundaries and pile-up intersections were observed to be strong barriers to slip band propagation. It was noted that slip propagation occurs by addition of dislocations to existing slip bands during single slip. In the case of multiple slip, they observed that slip propagation occurred by nucleation of new slip bands. This is because, when a second slip band intersected an active slip band, the active slip band was stopped. Microstructural studies were conducted on high nitrogen austenitic steels by Müllner et al [42] and Kubota et al [38] to understand the observed improved work-hardening properties due to the addition of nitrogen. Müllner et al [42] observed formation of multipoles and twinning after deformation. They suggested that multipoles provide work-hardening at smaller strains, but these structures could be unraveled at higher stresses and suggested that nitrogen promotes twinning and that is the primary reason for the observed high work-hardening at larger strains. In contrast, Kubota et al [38] did not observe any twinning in their alloys. They suggested that multipole formation provides work-hardening at smaller strains and at higher strains intersection of planar slip bands lead to formation of Lomer-Cottrell locks (a/6<110> dislocations) immobilizing the slip bands. For further propagation of slip, new slip bands had to be nucleated causing high work-hardening in these alloys. Thus, immobilization of the slip bands after intersection was thought to cause the observed high work-hardening rates.
In the case of titanium alloys there is no tendency to form multipoles as is evident from the deformation microstructure presented in section 6.3.2. Most of the microstructure consists of single dislocation pile-ups. This is very different from the deformation microstructures observed in the FCC-SRO alloys. Dislocations forming the multipoles in FCC-SRO alloys are usually of edge or mixed character. In the case of Ti-6Al it was shown that because of the large difference in the mobility of the edge/mixed versus screw segments, line lengths of edge/mixed segments will be relatively small. Hence, the chances of two edge/mixed bands of opposite sign meeting on parallel planes is low. Clearly, the deformation microstructure consists of long lengths of screw segments. Screw bands of opposite signs can meet on parallel planes, an example of which is shown in figure 6.21. Even in this case there is no tendency to form multipoles. The reason for this might be the large Peierls stress on the screw segments. This requires a large applied stress to move the screw segments anyway. But such interactions do cause difficulty in the slip band propagation. This is illustrated in figure 6.21b. Here two slip bands, which are less mature, are interacting on parallel planes. Band A has 11 dislocation pairs, i.e. positive (P) and negative (N) dislocations. The extrapolated location of the dislocation source has been indicated as S. All the 11 negative dislocations can be seen and only last 7-8 positive dislocations can be seen in band A. The dislocations that are present in band B (that can be seen in the image) are all of the same sign (P) and are moving to the left. One can observe that the negative dislocations in band A are experiencing difficulty in moving due to the interaction with the positive dislocations in band B. This can be inferred because all the positive dislocations of band A have moved much farther away from the location of the source, S. Further, the lead four dislocations of band B have moved farther away from rest of the dislocations in the slip band. This indicates that even
dislocations in band B which has many more dislocations than band A, experiences difficulty when passing the negative dislocations of band A. To reiterate, formation of dislocation multipoles, which could provide significant work hardening is not observed in titanium alloys. But, significant interaction between parallel slip bands of opposite signs do occur, as discussed above, when slip bands are nucleated on relatively closely spaced parallel planes.

Another consequence of the observed planar deformation structures is that elastic interaction between dislocations is significantly reduced, because of the large distances between slip bands (certainly at smaller strain levels). Therefore, significant dislocation interactions can occur only when dislocation pile-ups intersect each other or when parallel slip bands are nucleated close to each other (an example of which was discussed above) or when slip bands interact with grain boundaries.

There is a lot of dislocation debris (dipole loops) present in the slip bands as can be observed in the deformation microstructures presented above (see for example figure 6.21b, the slip band below band B). Work hardening within the slip band is possible because of the interaction of dislocations with the dipole debris that are formed in the slip plane. These dipole loops could have been formed by cross-slip events or from pinching-off of jogs formed due to intersections. Since the debris loops present are resolvable in conventional bright field microscopy, it indicates that they are relatively large and are most probably formed due to cross-slip events. Formation of dipole-debris loops by cross-slip events is schematically illustrated in figure 6.22(a)-(c). These dislocation debris provide a barrier to dislocation motion due to their elastic interactions, which can slow down dislocation motion. This is schematically illustrated in figures 6.22(d). And figure
6.23 shows actual interaction between debris loops and trailing dislocations in slip bands in a SQ-600C-24h-400C-5h sample. Three parallel prism slip bands with \( a/3[2110] \) dislocations are shown here. Debris loops presumably formed by cross-slip events are present in all three bands (clearly seen in band C). There is significant interaction between the debris loops and the trailing dislocations in the slip bands, esp. in bands A and B. But the dislocations seem to pass through and eventually become straight. Both elastic interaction of parallel slip bands and debris loops with dislocations in the slip band clearly affect the dynamics of pile-up propagation, but they do not appear to stop dislocation motion.

Dislocation intersections are probably the main work hardening mechanism within the grains in the case of titanium alloys. In the literature, Blackburn and Williams [1] have reported that when two \(<a>\) type dislocation bands intersect, they could react and form a glissile band instead of forming a lock, according to \( a_1 + (-a_2) = a_3 \). In our work, formation of such glissile bands were not observed. Figure 6.11a shows the intersection between a prism slip band \( a/3[11\bar{2}0](\bar{1}000) \) (labeled A) and a basal \( a/3[\bar{1}2\bar{1}0](0001) \) (labeled B) in an IWQ-350C-100h sample. There is no apparent interaction between the two bands and they seem to pass each other without much difficulty. Intersection between two prism bands \( a/3[2110](01\bar{1}0), \) band A and \( a/3[\bar{1}2\bar{1}0](10\bar{1}0), \) band B in a SQ600C-24h-400C-5h sample is shown in figure 6.24. In this view, slip band B is invisible (there is residual contrast) and the slip plane is edge on. Clearly, dislocations seem to interact much more strongly in this case, but they still pass through each other. The slip bands remain planar even after such intersection processes. There are instances when slip band intersections do seem to provide significant work hardening as can be seen in figure 6.25. Here intersections between several different slip systems and several slip bands in a SQ-600C-
24h-400C-5h sample is shown. Slip bands labeled as A belong to \(a/3[\overline{2}1 \overline{1} 0](01 \overline{1} 1)\) slip system, band B to \(a/3[\overline{2}110](0110)\) prism system, bands labeled C to \(a/3[\overline{1}2 \overline{1} 0](1010)\) slip system and band D to \(a/3[\overline{1}2 \overline{T} 0](0001)\) system. In the basal slip band, D, the dislocations seem to be moving from the bottom of the image to the top. They appear to pass through many bands with little disruption. Similarly, the prism slip band B, seems to retain its planarity even after intersecting many prism slip bands C on the right side of the image and the basal band D. Same is true for the pyramidal bands A, which seem to retain their planarity even after cutting through many prism bands C and the basal band D. (Note: dislocations in system A are moving from right to left). But there is significant disruption of planarity in slip bands A on the left side, after the intersections. This could be due to significant cross-slip of the dislocations in these bands. Such cross-slip is also apparent in band B at the tip of the band, after the intersections. This suggests that such cross-slip is a response to the local stress state and may not be a consequence of the intersection process itself. Slip bands of system C seem to experience the most difficulty from the intersection processes. There is clearly significant disruption of planarity and generation of debris loops near the intersections. All the above indicate that slip band intersections do cause work-hardening. This is also supported by slip line evolution study which is discussed in section 6.3.5. But an important point to note is that no dislocation reactions that forms sessile dislocation locks are observed at slip band intersections. This implies that slip band intersections will certainly slowed down the propagation of slip bands, but they would not be stopped in many cases.

A qualitative mechanistic description of dislocation intersections will be made below to explain some of the observed behavior. Before this is discussed, a brief discussion of some basic dislocation properties in hcp metals is made below. The most basic property
of $\langle a \rangle$ type slip in hcp metals is that the three slip vectors are co-planar. This is fundamentally very different from the cubic systems and could have a significant impact on the work hardening characteristics of these alloys. Co-planarity of the slip vectors implies that when dislocation intersections between $\langle a \rangle$ type slip vectors occur, the dislocations on the basal plane will only have kinks due to such intersections. Hence, such intersections will not stop the dislocation propagation on basal slip planes. Further, co-planarity also dictates that the jogs formed due to intersection processes will be on the basal plane for $\langle a \rangle$ type dislocations gliding on either prism or pyramidal planes. Such jogs on edge or mixed dislocations will be glissile and provide very little resistance for further motion, if (and only if) there is a significant applied stress on the basal plane.

Based on the observed deformation microstructure, intersections between screw pile-ups of $\langle a \rangle$ type dislocations are the most common dislocation intersections. Considering such intersections between the basal, prism and pyramidal $\langle a \rangle$ bands, one can identify two types of intersections due to the limited slip systems. First kind is the intersection between a basal slip band and a prism slip band. Since, the basal and prism planes are orthogonal to each other, there are two different possibilities for the intersection. If the prism band is established first and is assumed to be quasi-stationary, then the basal band will interact only elastically and most probably pass between the dislocations in the prism band without cutting them. Such a process will not be very difficult because the dislocations are in a pile-up configuration and the distances between the dislocations in the slip band (on the prism plane) is fairly large as seen from the pictures shown before. In such a process, no work hardening occurs on either slip plane. The second possibility in prism-basal intersection, is when a basal band is established first and is stopped. When the prism dislocations approach the basal band, they will have to intersect at many points.
along the basal band, in order to pass through the band. This dislocation cutting process is illustrated in figure 6.26. When dislocations intersect, as discussed above, kinks will be formed on the basal dislocations and the prism dislocations will be jogged with the jogs lying on the basal plane. After such an intersection process, dislocations on the prism planes will be highly skewed (as shown in figure 6.26b). Let us consider the case of the lead dislocations in the prism band and a dislocation in the middle of the band. In the absence of SRO, the lead dislocation will bow between the pinning points due to the jogs and can expand on many different slip planes. In such a process, the slip band will become diffuse after the intersection process. When there is SRO, the dislocations will have to cut through SRO domains on parallel planes. This could be a difficult process. The dislocation segment lying on the original slip plane will preferentially be able to cut through the SRO domain because it can be assisted by the self stresses of the dislocations in the pile-up, which will be maximum on this plane. Due to motion of that segment, the line tension and the applied stress on the basal plane can assist in the lateral motion of the jogs. This process will help the dislocation to straighten itself on the original glide plane. The trailing dislocations will be biased to continue on the original glide plane, because of the glide softening that has taken place in the original slip plane. The slip band will remain planar even after such an intersection process due to the presence of SRO. Stronger the SRO the greater the probability of maintaining planarity after such intersection processes. In general, prism slip will be the primary slip system in many cases. The prism slip bands will be well developed and basal bands will probably avoid cutting the prism band. This seems to be the case in the IWQ-350C-100h sample shown in figure 6.11a. Hence, basal/prism band intersections may not provide significant work-hardening in many instances.
An example of the second type of intersection that occurs, is between two prism slip bands of different burgers vector. Again we are considering intersection between two bands of screw dislocations. In this case, the intersection process is very different, this is illustrated in figure 6.27. Here, both bands hit at a 60° angle to each other, and will have to cut through the dislocations in the other band sequentially. This is a very different process when compared to the intersection between a prism and a basal band, where the prism dislocations intersect the basal band at many locations. When such intersection occur at low strains, the slip band can bow around the intersecting slip band and continue to propagate without cutting through. This process is schematically illustrated in figure 6.28. Such a process is possible because there will be large dislocation free channels between the slip bands in the intersecting plane. Possible operation of such a mechanism is shown in figure 6.21b (see the slip band above band A). There are two prism slip bands intersecting. The a/3[T2T0](1010) is almost edge on in this view and is invisible. The other prism band a/3[2110](0110) can be seen intersecting the first slip band and there are long lengths of edge/mixed segments that are present at the intersection indicating that the bow around process could be occurring in this case.

Slip band intersections that were shown in figures 6.24 and 6.25 show that the dislocations on intersecting slip bands are able to cut through each other and continue to propagate, not bow around the bands as suggested above. At the present time, the exact mechanism by which these dislocations are able to cut through each other and propagate is not clear.

Finally, it should be mentioned that intersection between a pyramidal \(<a>\) type slip band and other slip bands on either prism or pyramidal slip planes will be similar to the
prism/prism intersection process, mechanistically. Clearly, the elastic interactions will be very different. But, the intersection between a pyramidal band and a basal slip band is different. Here the pyramidal band will be cutting the basal band at many locations at once (similar to the prism/basal interaction). And the basal slip band will be intersecting the dislocations on the pyramidal band sequentially (similar to the prism/prism interaction).

Intersections between \(<a>\) type slip bands on different slip planes was discussed so far. Since \(<c+a>\) dislocation activity is observed in these samples, interaction between \(<a>\) type slip bands and \(<c+a>\) dislocations is also observed. An example of which is shown in figure 6.29. Even in this case the \(<a>\) dislocations seem to continue to propagate after intersecting \(<c+a>\) dislocations. One can observe a lot of dislocation debris after such intersections, but again the dislocations seem to straighten themselves as they continue to propagate. Unlike intersections between \(<a>\) slip bands, in many cases intersection between \(<c+a>\) dislocations and \(<a>\) dislocation slip bands do provide significant work-hardening.

To summarize, prism/prism and prism/basal \(<a>\) type slip band intersections are the most important because they are the most common slip systems that are operative. As noted above, slip band intersections between basal and prism bands do not seem to cause significant work-hardening in many cases. In most cases prism and basal slip bands retain their planarity even after intersections. An attempt was made to identify some of the mechanisms by which slip bands can retain their planarity after intersections as well as to identify possible work-hardening mechanisms. The exact nature of the intersection processes between prism/prism, prism/pyramidal and basal/pyramidal is not very clear at
this point. But, intersections do cause some work-hardenings as have been observed in some cases and in the slip evolution study discussed in section 6.3.5. Two slip bands reacting to form a third glissile slip band was not observed in our studies. Intersecting slip bands forming sessile products, which could stop slip band propagation was not observed either.

Since deformation propagation is through slip bands, the interaction of these bands with grain boundaries is very important. Examples of slip band interaction with a grain boundary is shown in figure 6.30. Grain boundaries seem to provide a strong barrier to slip propagation, because the dislocations in the slip band are piled-up against the boundary. It is interesting to note that the dislocations are in a mixed character near the grain boundary as shown in figure 6.30a (in a SQ-600C-24h-400C-5h sample). Whereas in figure 6.30b, the dislocations are predominantly in screw character (in an AC-I sample). This is because the dislocations seem to align themselves along the boundary plane near a grain boundary. It can be seen in figure 6.30a, far away from the boundary, the dislocations are in the screw orientation. It was mentioned in section 6.3.3 that the dislocation core of \(<a>\) type screw dislocations is non-planar. Therefore, the stress required to move the screw segments will be very high, i.e. the Peierls stress will be large for the screw segments. Because the dislocations tend to orient themselves along the grain boundary plane, the number of dislocations that are in mixed character in the slip band, will change with the angle the screw direction makes with the grain boundary plane. This has a significant impact on the internal stress distribution at the grain boundaries due to even a single slip band. The stress concentration will be maximum at the boundary location where the dislocations in the band are in edge character and will be minimum at the boundary where the dislocations in the slip band are in screw character,
and intermediate at the other boundaries, which will depend upon the number of mixed dislocations in the band. This is because, Peierls stress is low for the edge/mixed orientation and high for the screw orientation. Hence, the efficiency of the pile-up in developing stress concentrations at the tip of the pile-up due to their self stresses will be maximum for an edge pile-up. Since slip is planar, this suggests that grain boundaries could provide significant strengthening. On the other hand, there is no tendency to form low energy dislocation structures like multipoles, in titanium alloys. This would imply that there will be large stress concentrations at the grain boundaries due to the slip bands. Hence, slip transmission/nucleation in the neighboring grains could be relatively easy in these alloys.

In the above discussion, some microstructural features were identified which seem to be responsible for the very low $n$ values in observed titanium alloys. No low energy structures like multipoles are formed in titanium alloys. Strain hardening within the slip bands is very low. Slip band intersection which seem to immobilize slip bands in FCC-SRO alloys do not seem to cause such immobilization in many cases. This would imply that work-hardening due to such intersections will be lower in the case of titanium alloys. All the above factors cause strain hardening within a grain to be low. The slip bands do interact strongly with grain boundaries and get stopped. But the large stress concentrations developed at the boundaries due to the slip bands can cause slip nucleation/transmission in the neighboring grains to be a relatively easy process. A combination of these factors seem to cause the observed low work-hardening rates in titanium alloys.
6.3.5. Slip line evolution study

Since plasticity occurs by the cooperative movement of large numbers of dislocations, direct insight into the overall progression of deformation processes occurring during creep can be obtained using conventional optical microscope observations. Such a study was conducted on a Ti-6Al-DI sample. Figure 5.5 shows that there are three regimes of exhaustion in the primary transient of a Ti-6Al-DI sample when deformed at 716 MPa. This is similar to the behavior of IWQ, IWQ-age and AC-age samples that were discussed in section 6.2. The deformation behavior of Ti-6Al-DI is also planar. This is shown in figure 6.31, where intersection between a prism and basal system is shown. It can be seen that slip is planar on both planes and the slip bands seem to pass through each other with little difficulty, similar to what was discussed in section 6.3.4.

Shown in figures 6.32-6.35 are a sequence of surface, slip-line observations on a Ti-6Al-DI sample crept at 716 MPa. The creep behavior of the slip line evolution study sample was similar to and representative of the monotonic creep samples. The creep test was interrupted periodically in order to perform slip trace studies. The slip line observations were made after creep times of 96 sec, 1000 sec, 9000 sec and $931 \times 10^3$ sec (258.5 hrs). After creeping for 96 sec, slip traces were observed in only some of the grains, presumably those most favorably oriented. An example is shown in the grain labeled as A in figure 6.32. In these favorably-oriented grains, only a small number of slip traces are observed, most of which completely traverse the deforming grains. It is not known conclusively whether the slip traces are due to individual dislocation arrays, or to packets of arrays, although the common observation of the latter in TEM studies would favor this conclusion. It should be noted that this first micrograph corresponds to the time regime for which the creep time exponent is initially small, indicating relatively rapid exhaustion.
of the creep rate. Since many grains exhibit only single slip activity at this point, the exhaustion may be due to interaction of dislocation arrays with grain boundaries, which provide the only strong obstacles to dislocation propagation.

At larger times (figure 6.33), dislocation arrays are clearly interacting commonly with grain boundaries. Three important features to note at this stage are: (a) intensification of existing slip traces, (b) the observation of new slip traces in already active grains, and (c) initiation of new slip traces in adjacent grains, in which slip had not been apparent previously. Feature (a) is particularly interesting since it does indicate that even when the very strong barrier of a grain boundary is encountered, slip can continue even locally. The intensification of the slip traces is probably due to the addition of more dislocation arrays to existing packets of arrays, again suggesting little ability to strain harden even at a local scale. Note also that these features are associated with that part of the creep curve exhibiting the largest time exponent (i.e. the smallest rate of exhaustion). The initiation of large numbers of new dislocation arrays, particularly at grain boundaries neighboring already active grains, may be responsible for the smaller exhaustion rates in this regime.

In figure 6.34, it can be seen that intense initiation of new slip traces near grain boundaries continues. However, it is interesting that only a small number of these slip traces have actually propagated large distances into the grains. Many of the slip traces are seen to terminate in the middle of the grains, indicating that their traversal through the grains is not very rapid. In fact, comparison of the apparent tips of the slip traces from one micrograph to the next indicates that the dislocation arrays or packets propagate very sluggishly. Slip traces associated with secondary slip systems are also apparent in many grains. One would expect more slip system interactions as more secondary slip is
activated. The region indicated as I in the figure shows one such interaction between primary and secondary slip systems. It can be seen that there are many more secondary slip lines below the primary slip lines. There are far fewer secondary slip lines above the interaction with the primary slip system. These are indications that slip system interaction inhibits the operation of secondary systems and may therefore contribute to the moderately increased rate of exhaustion in this time regime.

Finally, in figure 6.35, it can be seen that slip trace activity is intense in both the grain interiors and at the grain boundary. The average distance between the slip traces has become very small. This would indicate that interaction between parallel slip bands are becoming more important, with extensive interaction between primary and secondary slip bands as well. All these factors could cause the increased rate of exhaustion and lead to the somewhat reduced time exponent observed at long times.

6.4 Strengthening due to SRO and estimation of $\gamma_{\text{SRO}}$ and $\tau_{\text{SRO}}$ in titanium alloys

It was discussed in section 2.5.1 that SRO does provide strengthening due to the creation of a diffuse APB ($\gamma_{\text{SRO}}$) on the slip plane when a dislocation shears an alloy with SRO. In the literature two different approaches have been taken to measure this quantity. One approach has been to estimate $\gamma_{\text{SRO}}$ theoretically from the number of unfavorable bonds that are formed across the slip plane due to passage of a dislocation, for a given SRO state[21, 22, 44-52]. All of these studies have been in fcc alloys except for the work of Pitsch[46, 47], which was on bcc alloys. The other approach has been to estimate the friction stress due to SRO ($\tau_{\text{SRO}}$) from the observed dislocation structures in a TEM foil[14, 15, 17-19, 53, 54]. All the dislocation studies have been on fcc alloys. A discussion of both approaches is given in section 2.5.1.

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Theoretical models for calculating $\gamma_{SRO}$ from measured SRO parameters is not available for hcp metals at the present time. In the case of hcp metals, $<a>$ type slip can occur on three crystallographically distinct planes namely, basal, prism and pyramidal planes. This would imply that even for the same SRO state, $\gamma_{SRO}$ will be different on the different slip planes even when one considers just $<a>$ type slip. If $\gamma_{SRO}$ is high enough to influence the deformation behavior, these differences could affect the choice of slip systems and deformation microstructure in these alloys. At the present time these differences have not been sorted out and their influence on deformation behavior is not known.

It was observed by Ogden et al[55] and Blackburn and Williams[1] that in Ti-Al alloys containing less than 10at% Al, the dependence of yield strength on aluminum concentration was much stronger than what one would expect for solid solution strengthening. Blackburn and Williams suggested that this could be due to SRO strengthening. Conrad[56] attempted to study strengthening due to SRO using the data of both Ogden et al and Blackburn and Williams. He used Flinn’s[44] model for FCC-SRO alloys, which related the SRO state to strengthening due to SRO, assuming nearest-neighbor pair interactions. Conrad assumed that slip occurred only on prism planes and made a further simplifying approximation that SRO is weak, in his calculations. He estimated the pair interaction energy from the mechanical test data based on these simplifying assumptions and compared it to theoretical estimates. Since, he found good agreement for pair interaction energies, it was suggested that SRO could be the main strengthener in dilute Ti-Al alloys(Al content $\leq$ 10at%). Although, Conrad’s analysis did indicate that there could be significant strengthening from SRO in Ti-Al alloys, he did not estimate this strengthening due to SRO. Further, his analysis treated SRO
strengthening to be a single quantity for a particular composition. Whereas, it was shown by Schwander et al.[50] that strengthening due to SRO will be dependent upon the SRO state in the material. And the SRO state present in the material will be dependent on the heat treatment that the sample has undergone.

In chapter 4 we presented neutron diffraction evidence that showed the presence of SRO in a Ti-6Al alloy. We are in the process of calculating Warren-Cowley SRO parameters for these alloys. Theoretical calculation of $\gamma_{\text{SRO}}$ from the observed SRO state is possible only after the SRO parameters have been evaluated. An attempt was made to estimate $\tau_{\text{SRO}}/\gamma_{\text{SRO}}$ from the deformation microstructure as Warren-Cowley SRO parameters are not available at the present time.

It was discussed in section 2.5.1 that in the literature $\tau_{\text{SRO}}$ has been evaluated from pile-ups immobilized in the middle of a grain using both post-mortem and in-situ TEM studies[14, 17-19]. In the case of Ti-6Al such a method is not directly applicable as will be explained below. An implicit assumption in evaluation of $\tau_{\text{SRO}}$ from pile-up structures is that the dislocations in the pile-up move in an athermal fashion under an applied stress. Put another way, the assumption is that the friction stress opposing the motion of the dislocation is only due to $\gamma_{\text{SRO}}(\tau_{\text{SRO}})$ and due to the elastic interaction with solutes($\tau_{\text{SS}}$). The lattice friction opposing dislocation motion $\tau_{\text{PEIERLS}}$ is assumed to be negligible. From force equilibrium of an immobilized pile-up one can then evaluate $\tau_{\text{SRO}}$ if one can evaluate $\tau_{\text{SS}}$ independently. In the works on FCC-SRO alloys, $\tau_{\text{SS}}$ has been evaluated from the friction stress experienced by the last dislocation( dislocation at the tail) in a pile-up[14, 18, 19]. Figure 6.36 shows a dislocation pile-up in a SQ-600C-24h-400C-5h sample. The dislocations in the pile-up are in screw orientation. Examination of the inter-
dislocation distances in the pile-up shows that after the first few dislocations the spacing between the dislocations are more or less constant. This is very different from a classical pile-up, where the inter-dislocation distances continually increase from the head of a pile-up to the tail due to the self stresses of the other dislocations in the pile-up. That the pile-ups observed in titanium alloys are non-classical in nature is illustrated in figure 6.36b. In this figure, the interaction stress on each dislocation in the pile-up due to self stresses of the other dislocations is plotted as function of distance from the tip. The dislocations were assumed to be straight and infinitely long along the screw orientation. The interaction stresses were calculated using the following expression,

$$\tau_{\text{int}}^i = \sum_{j \neq i}^{n} \frac{\mu b}{2\pi} \frac{1}{x_i - x_j}$$  \hspace{1cm} (6.1)$$

where, $\mu$ is the shear modulus, $b$, the burgers vector, $x_i$ the position of dislocation $i$ in the pile-up and $\tau_{\text{int}}^i$ is the interaction stress experienced by dislocation $i$ due to all the other dislocations in the pile-up. A value of 46.2 GPa[57] was used for the shear modulus and 0.293 nm for $b$[58]. Interaction stresses due to dislocations outside the pile-up are ignored. The inter-dislocation distances were measured from the center of the slip plane in the TEM micrographs, correcting for the projected distances. Even though some of the dislocations are curved, they were assumed to be straight. Figure 6.36b shows that after the first few dislocations the interaction stresses on the dislocations in the pile-up are not zero. It oscillates and can be a significant fraction of the stresses at the tip of the pile-up. All the above indicate that even the dislocations in the middle of pile-up experience a large friction stress opposing their motion. We discussed in section 6.3.2 that $<\alpha>$ type dislocations in titanium alloys have a non-planar core structure along the screw orientation. Hence, the screw dislocations experience a large lattice friction stress, $\tau_{\text{Peierls}}$ opposing their motion. Since the quantity $\tau_{\text{Peierls}}$ is not very well defined in our case, we
cannot estimate the friction due to SRO, $\tau_{\text{SRO}}$ from static pile-up calculations using arguments used in FCC-SRO alloys.

In order to evaluate the friction stress due to SRO we have taken a different approach, but still using the observed dislocation structures. Figure 6.37 shows "hair-pin" like structures where part of a dislocation loop is shown with both the edge and screw segments. The dislocation loop is elongated in the screw orientation as one can see from the neighboring screw pile-ups. Such observations have been made in AC, SQ-600C-24h-400C-5h and IWQ-350C-100h samples. Observation of such "hair-pin" structures is infrequent, because the edge dislocations are lot more mobile than the screws. These elongated loops have been observed usually in pairs. No more than two dislocations have been observed in such configurations. Presence of such "hair-pin" like structures provides an important clue regarding the deformation characteristics of these alloys. In the case of FCC-SRO alloys which were discussed in section 2.5.1, the friction stress opposing dislocation motion was observed to be at least twice as high as the applied stress[14, 17-19]. Hence, it was argued that dislocation motion was possible only in the form of dislocation pile-ups. Once, a critical pile-up is established, because of the glide plane softening effect, the trailing dislocations will assist the leading dislocation in overcoming $\tau_{\text{SRO}}$ by means of their interaction stresses. Observation of "hair-pin" like structures indicate that individual dislocations are mobile under the applied stress in the case of Ti-6Al alloy, i.e., the applied stress is above $\tau_{\text{SRO}}$.

Formation of the "hair-pin" structure is envisioned as follows. Since, the edge segments are lot more mobile than the screws, beyond a critical separation distance ($L_c$) between the screws, the edge segment will run out into the grain laying out a screw dipole in the
process. This process is very similar to the constrained motion of misfit dislocations in a thin film epitaxial layers\[^{59}\]. The critical stress (\(\tau_c\)) to be overcome before the edge dislocation can run-out unconstrained can be calculated using the following expression,

\[
\tau_c = \frac{\mu b}{2\pi L_c} \ln\left(\frac{L_c}{b}\right)
\]

(6.2)

where, \(\tau_c\) is the critical stress needed to lay out the screw dipole, \(\mu\) is the shear modulus (46.2 GPa), \(b\) is the burgers vector and \(L_c\) is the critical separation distance. Figure 6.38 shows schematically the creation and alteration of \(\gamma_{\text{SRO}}\) as a function of the number of dislocations shearing the slip plane. The lead dislocation creates a diffuse APB of energy \(\gamma_{1\text{SRO}}\) and the second dislocation heals the diffuse APB only partially and lowers its energy to \(\gamma_{2\text{SRO}}\). The second dislocation decreases the diffuse APB energy only partially because it is only short-range order. With further dislocation motion there could be small oscillations of the energy of the APB. After the first few dislocations the trailing dislocations in the slip band do not alter the diffuse APB energy, therefore, do not experience any friction from the diffuse APB present on the slip plane. Figure 6.39 shows the forces acting on the two edge segments forming the “hair-pin” structures due to the interaction stress and friction from the two diffuse APBs. If one considers the motion of the pair of edge segments (as super-dislocation motion), it shows that the dislocation pair experiences a friction stress due to \(\gamma_{2\text{SRO}}\) opposing their motion. From the micrographs one can measure \(L_c\) for the dislocation pair of the “hair-pin” structures. Using that one can calculate \(\tau_c\), the minimum stress needed to move the pair from equation 6.2. At the critical separation distance, the effective stress on the dislocation \(\tau_{\text{eff}} (= \tau_{\text{app}} - \tau_f)\) will be equal to \(\tau_c\), where the friction stress, \(\tau_f = \tau_{\text{SRO}} + \tau_{\text{SS}}\). Hence, one can calculate \(\tau_f\) from the measured \(\tau_c\), if the applied stress \(\tau_{\text{app}}\) is known. In addition to the “hair-pin” structures, such measurements can be made from the separation distance of screw super-dipoles.
which have been laid out by the above process. The foil normal for the grains from which
the Lc measurements were made was determined. This was taken to be the stress axis for
that grain and the schmid factor for the observed slip system was calculated. From this
the applied stress \( \tau_{\text{app}} \) on the slip plane was calculated. The friction stress (\( \tau_f \)) from
measured distances Lc have been calculated for IQW-350C-100h, AC samples as well for
SQ-600C-24h-400C-5h samples. These values are listed in table 6.10. In all cases the
friction stress measurements have been made on the prism plane. From the estimated
friction stresses, one can get the friction due to \( \gamma_2^{\text{SRO}} \) if we know the friction due to the
solute(s)\( \tau_{\text{ss}} \). The estimated diffuse APB will be an upper bound because the edge
segments in the “hair-pin” structures were mobile under the effective stress experienced
by them.

In FCC-SRO alloys \( \tau_{\text{ss}} \) has been measured from the friction stress experienced by the
dislocation in the tail of the pile-up. It was discussed above that \( \tau_{\text{Peierls}} \) on the screw
dislocations comprising the pile-up is very high in the case of Ti-6Al. Hence, an
evaluation of \( \tau_{\text{ss}} \) from the friction stress on the last dislocation in the pile-up is not
feasible in our case. In the literature, it has been shown that aluminum strengthens
titanium mainly in an athermal fashion[60, 61]. From polycrystalline studies it has been
noted that the strengthening due to aluminum in binary Ti-Al alloys has a linear[8, 60] or
greater than a linear dependence[1, 55, 56] as a function of solute content below 13at%
Al. These dependencies suggest that the strengthening of titanium by aluminum is greater
than what one would expect from conventional solid solution strengthening theories, as
discussed above. Sakai and Fine[5] observed work softening during prism glide of a Ti-
5.2at% Al alloy and suggested that the observed work softening is due to the presence of
SRO. To evaluate \( \tau_{\text{ss}} \) as accurately as possible and to eliminate possible strengthening

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contribution due to SRO, $\tau_{SS}$ has been evaluated using data of dilute Ti-Al alloys ($<2.5\text{at}\%\text{ Al}$). Two different methods were employed to estimate $\tau_{SS}$. Single crystal CRSS data from the work of Paton et al[62] for Ti-0at% Al and Ti-2.5at%Al as well as data for Ti-2.1at%Al from Sakai and Fine[5] were plotted as a function of $C_{ij}^{\frac{1}{2}}$. From this plot the extrapolated $\tau_{SS}$ for a Ti-6Al(10at%Al) alloy was estimated to be 69MPa. Okazaki et al[61] have evaluated the athermal strengthening due to Al in dilute Ti-Al alloys with Al content upto 2at% Al. Extrapolating their plot to Ti-6Al, we obtained a $\tau_{SS}$ in polycrystals to be 156 MPa. Using a Taylor factor of 5[63], $\tau_{SS}$ for prism slip was estimated to be 31 MPa. Using these two estimates of $\tau_{SS}$, $\gamma_{1}^{SRO}$ was evaluated for all three samples and are listed in table 6.10. The estimated $\gamma_{2}^{SRO}$ values are between 25 to 48 mJ/m$^2$. Comparing these values with those that have been evaluated for various FCC-SRO alloys with strong SRO[15, 18, 50], which range from 14 to 32 mJ/m$^2$, the estimated energies for Ti-6Al are very reasonable.

The estimated values of $\gamma_{2}^{SRO}$ suggests that there is not a significant difference between the SRO state in an AC sample versus that in a SQ-600C-24h-400C-5h sample. From the observed deformation microstructure, there are differences between the two samples. Pairing of lead dislocations in the screw pile-ups has been observed only for the first two dislocations in the case of an AC sample, whereas at least the first 4 and frequently the first 6 dislocations have been observed to be paired in the case of the SQ-600C-24h-400C-5h sample. Macroscopically, there is a large difference in the initial creep response between the two heat treatments, suggesting there could be a difference in the SRO states. Force balance of the lead edge segments in the "hair-pin" configurations shown in figure 6.39, shows that to a first approximation, the equilibrium separation distance between two segments will be determined by the difference between $\gamma_{1}^{SRO}$ and $\gamma_{2}^{SRO}$, diffuse APB
energies. Smaller the difference between the two energies, larger will be the separation between the leading edge segments. From the “hair-pin” structures presented in figure 6.37, one can see that there is a significant difference in the configuration between the AC sample and that present in the SQ-600C-24h-400C-5h sample. In the case of the AC sample the leading edge segment of the pair is not present in the foil. This suggests that the separation distance between the two segments could be as large as the distance between the second segment and the foil surface. This implies that difference in the energies \( \gamma_1^{\text{SRO}} \) and \( \gamma_2^{\text{SRO}} \) is relatively small in this case. Whereas in the case of SQ-600C-24h-400C-5h sample, the pairing is relatively strong. This can also be seen in figure 6.19, where a dislocation source is shown. Here all the leading edge segments appear paired. From the pairing of the leading edge segments shown in figure 6.37, the difference between the two diffuse APB energies, \( \gamma_1^{\text{SRO}} - \gamma_2^{\text{SRO}} \) was estimated to be 6mJ/m\(^2\). Difference between \( \gamma_1^{\text{SRO}} \) and \( \gamma_2^{\text{SRO}} \) has also been evaluated in FCC-SRO alloys[15, 18, 50] and is between 1-3 mJ/m\(^2\), which is similar to our estimates. The above discussion suggests that in the case of AC sample \( \gamma_1^{\text{SRO}} \approx \gamma_2^{\text{SRO}} \), but in the case of the SQ-600C-24h-400C-5h sample \( \gamma_1^{\text{SRO}} = \gamma_2^{\text{SRO}} + 6\text{mJ/m}^2 \). Such measurements were not made on the IWQ-350C-100h sample, where the distance between leading edges could not be determined unambiguously.

The above discussion shows that in the case of Ti-6Al the applied stresses are higher than the friction stress due to SRO(\( \tau_{\text{SRO}} \)), because in these alloys, deformation is controlled by the motion of screw dislocations. Hence, the applied stresses have to be relatively high in order to move the screw dislocations because they experience a large Peierls stress.
In the studies on FCC-SRO alloys, it was argued that dislocation motion occurs in pile-ups because the friction stress opposing dislocation motion was higher than the applied stresses. Since, the applied stress is higher than \( \tau_{sro} \) in the case of Ti-6Al, we need to understand the reasons for the formation of dynamic screw pile-ups. Based on the discussion of dynamics of dislocation pile-up formation in section 2.5.2, it was noted that calculations showed that when dislocations were emitted from a source, they tend to form an "inverse" pile-up against the source[64-70]. Such "inverse" pile-ups against a grain boundary dislocation source has been observed experimentally in metals by Murr[71]. "Inverse" pile-ups were rationalized on the basis that the self stresses of the trailing dislocations in the pile-up assist the motion of leading dislocations, whereas the self stresses of the leading dislocations retard the trailing dislocations in the pile-up. The formation of an "inverse" pile-up against the source was observed to hold for many different velocity-stress laws and even in the case of thermally activated motion of dislocations through an array of short-range barriers[68]. It was also true even when there was a uniform friction stress opposing dislocation motion. All these factors affected the dynamics of pile-up formation, but the shape of the pile-up was an "inverse" pile-up against the source. In our observations the dynamic pile-ups are of the normal kind, i.e. the dislocations are piled-up away from the source.

In order to better understand the dynamics of screw pile-up formation, a velocity law was derived for thermally activated motion of a screw dislocation in the presence and absence of \( \gamma_{sro} \). Dislocation motion was assumed to be accomplished by kink-pair formation and migration. The screw dislocations were assumed to form abrupt kinks of edge character of height \( h \) and separation \( l \) (figure 6.40). Kink pair formation process is thermally activated, but the kink pair migration was assumed to be athermal. These assumptions
were motivated by the fact that they provide a direct physical link with our observation that the edge segments are much more mobile and their velocities are much higher compared to the screws. Further, in-situ deformation in a HVEM showed the screw motion to be very viscous and continuous in Ti-6Al. This observation is different from the “jerky” glide that was observed in α-Ti by Farenc et al[31] and has been attributed to the “locking-unlocking” mechanism.

Stress dependence of the activation energy for the kink pair formation is shown below,

\[ F(\tau) = 2F_k \left[ \left( \frac{\mu b^2 h^2}{8\pi l} \right) \left( \frac{1+\nu}{1-\nu} \right) \right] - \tau bh - \gamma_{SRO} hl \]  \hspace{1cm} (6.3)

where \( \mu, b, v \) have the usual meaning, \( F_k \) is the self energy of an edge kink, \( \tau \) is the applied stress on the dislocation, \( h \) and \( l \) are the kink height and kink separation distance respectively. The first term on the right hand side of equation 6.3 is the self energy of two edge kinks, second term is the elastic interaction energy between the two edge kinks (taken from Hirth and Lothe[72], p 244), third term is the work done by the applied stress and the last term is the energy of the diffuse APB created when a kink pair is formed in an alloy with SRO. The term with \( \gamma_{SRO} \) will not be present in the absence of SRO, or in our case in the kink formation energies of the trailing dislocations in a pile-up, because they do not alter the diffuse APB that has already been created by the lead dislocation(s).

One can then derive the equilibrium double kink separation distance, \( l^* \) as a function of effective stress on the dislocation from the maximum value of \( F(\tau) \), which is shown below.

\[ l^* = \left[ \left( \frac{\mu bh}{8\pi \tau} \right) \left( \frac{1+\nu}{1-\nu} \right) \right]^{1/2} \]  \hspace{1cm} (6.4)
Equation 6.4 gives the equilibrium separation distance as a function of stress for the case where no diffuse APB is created, this is the same expression derived by Hirth and Lothe (see p.541)[72]. Whereas, equation 6.5 gives the equilibrium separation distance as a function of stress in the case where a diffuse APB is created. This equation demonstrates that the creation of a diffuse APB can be treated as a lowering of the stress assistance available for the kink pair formation process.

Substituting the equilibrium separation distance, \( l^* \) back into equation 6.3, one can get the activation energy for a kink pair with equilibrium separation, which is shown below for the two cases with and without \( \gamma_{SRO} \).

\[
F^* = 2F_\tau - \left[ \frac{\mu b^2 \tau}{2\pi} \left( \frac{1 + \nu}{1 - \nu} \right) \right]^{1/2} \tag{6.6}
\]
\[
F^* = 2F_\tau - \left[ \frac{\mu b^2 \tau}{2\pi} \left( \frac{\tau - \gamma}{b} \right) \left( \frac{1 + \nu}{1 - \nu} \right) \right]^{1/2} \tag{6.7}
\]

Using an approach similar to Dorn and Rajnak[73], we have derived the kink nucleation rate to be,

\[
N_\kappa = \nu \frac{L}{l} e^{-\frac{F^*}{kT}} \tag{6.8}
\]

where, \( kT \) has the usual meaning, \( \nu' \) is the attempt frequency (see Kocks et al[74] for a discussion), \( L \) is the length of dislocation segment available for kink pair formation and
L/l is the number of available sites for kink pair formation and F* is the activation energy. Dom and Rajnak have also shown that at steady state, the kink pair nucleation rate and the kink pair annihilation rate are equal. In other words, there is only one kink pair present over a dislocation segment of length L, at any given time, or a kink pair will be nucleated when a kink pair is annihilated over a segment of length L.

\[ A_k = \frac{2v_k}{L} = \sqrt{\frac{L}{l}} e^{-\frac{F^*}{kT}} \quad (6.9) \]

where \( A_k \) is the kink pair annihilation rate and \( v_k \) is the kink velocity. From this one can get a relation for the segment length \( L \) as,

\[ L = \left( \frac{2v_k l}{\sqrt{v}} e^{-\frac{F^*}{kT}} \right)^{1/2} \quad (6.10) \]

Substituting \( L \) back into kink pair nucleation rate (equation 6.8), one can then get the dislocation velocity law to be,

\[ v = \frac{v}{h} \left( \frac{2v_k}{l} e^{-\frac{F^*}{kT}} \right)^{1/2} \quad (6.11) \]

Assuming the kink motion is athermal and the stress dependence of kink velocity is of the form,

\[ v_k = \frac{t_b}{B} \quad (6.12) \]

where, \( B \) is the drag coefficient. And substituting for the equilibrium separation distance, the dislocation velocity is given by,

\[ v = M \tau^{3/4} e^{C\tau^{1/2}} \quad (6.13) \]

where \( M = \left( \frac{v}{B} \right)^{1/2} 2^{5/2} \left[ \frac{1 - \nu}{1 + \nu} \right]^{1/4} \left[ \frac{\pi b^3}{\mu} \right]^{1/4} e^{-\frac{F_k}{kT}} \), \( C = \left( \frac{\mu b^3 h^3 \left( 1 + \nu \right)}{2\pi \left( 1 - \nu \right)} \right)^{1/2} \), and \( \tau' = (\tau_{app} - \tau_s) \).
in the absence of $\gamma_{SRO}$ and $\tau' = \left( \tau_{\text{app}} - \tau - \frac{\gamma_{SRO}}{b} \right)$ in the presence of $\gamma_{SRO}$.

Figure 6.41 shows the effect of $\gamma_{SRO}$ on the dislocation velocity. This is a plot of normalized dislocation velocity $\frac{\nu}{M}$ versus the (effective) stress for various values of $\gamma_{SRO}$. The constant $C$ in equation 6.13 was evaluated with the following parameters, $\mu=46.2$ GPa, $h=c=0.468$ nm, $b=a=0.293$ nm, $T=300$ K and $\nu=0.319$. The stress level range from 0-1000 MPa and $\tau_{SRO}$ range from 0-200 MPa; this covers a range wider than the applied stress levels of interest and reasonable values of $\gamma_{SRO}$. Figure 6.42 shows that the dislocation velocity at a given stress decreases with an increase in $\gamma_{SRO}$. The plot also shows that there is a critical stress ($\gamma_{SRO}/b$) below which the dislocation will not move, i.e. the dislocations will not move if the stress is not high enough to overcome $\tau_{SRO}$. This behavior is not captured in equation 6.13 and has been artificially imposed in the plots.

But, based on the FCC-SRO work, the dislocations will be able to move in the form of pile-ups at a stress lower than the critical stress ($\gamma_{SRO}/b$). In our case, where the applied stress is above $\tau_{SRO}$ the plot shows that at a given effective stress ($\tau_{\text{eff}} = \tau_{\text{app}} - \tau_{SS}$), the lead dislocation(s) in the slip band will be moving slower than the trailing dislocations. This is because, the trailing dislocations in the slip band do not experience any friction due to $\gamma_{SRO}$, since they do not alter the $\gamma_{SRO}$ created by the leading dislocations. In order for the slip band to propagate at a constant velocity at a given stress, the trailing dislocations will have to pile-up against the leading dislocation(s). This is in agreement with the calculations of Arsenault[75] and Olfe and Neuhäuser[14], which showed that dynamic pile-ups will be formed only when there is a glide plane softening effect.
If one makes the simple assumption that except for the lead dislocation, the rest of the dislocations in the slip band move on an average at a velocity corresponding to $\tau_{\text{eff}} = \tau_{\text{app}} - \tau_{ss}$ given by equation 6.13. Then the stress amplification needed on the lead dislocation, to move it at the same velocity as the rest of the dislocations in the slip band, is equal to $\tau_{\text{SRO}}$. If one assumes that the configuration of the screw dislocation pile-ups (i.e. the positions of the dislocations in the pile-up) was not altered during unloading and subsequent preparation of the thin foil for TEM analysis. (Such an assumption is reasonable in our case because there is a large Peierls stress on the screw dislocations and there is no significant backflow after a complete load drop (see section 6.5)). The stress amplification on the lead dislocation can be calculated from the dislocation positions measured from TEM micrographs using equation 6.1. Again, the dislocations were assumed to be straight and infinite along the screw orientation. The inter-dislocation distances were measured and corrected for their projections. Interaction stresses from other bands in the thin foil were neglected. Pile-up tip stresses from a few isolated single ended and double ended pile-ups are listed in table 6.11. It also lists the number dislocations in the pile-up. Calculated pile-up stresses are less than half of the applied stress. This is very different from the pile-up tip stresses estimated in FCC-SRO alloys[14, 17-19], where it has been estimated to be greater than twice the applied stresses. This again supports our conclusion that the applied stress is above $\tau_{\text{SRO}}$, in our case. The estimated $\gamma_{\text{SRO}}$ values from equating the tip stress to $\tau_{\text{SRO}}$ are also listed in table 6.11. These values are similar to what was estimated from the calculations using the “hair-pin” like structures and are comparable to those estimated in FCC-SRO alloys.

At this point it worth discussing some of the assumptions that have been made for evaluating $\gamma_{\text{SRO}}$ from the pile-up structures present after deformation. One of the main
assumptions was that the slip band as a whole moved at a constant velocity at a given stress. In-situ deformation experiments in a model alloy representing the γ phase of a Ni-based superalloy by Jouiad et al[19] showed that the slip bands indeed move at a constant velocity. This is also supported by the calculations of Olfe and Neuhauser[14], who stated that the velocity of a dynamically piled-up group is determined by the applied stress and the configuration of the piled-up group will not be altered by the applied stress. Arsenault[75] performed computer simulations of dislocation motion in a neutron irradiated material. Dislocations were assumed to move in a thermally activated fashion among short-range and long-range barriers. The lead dislocations were assumed to remove the short-range barriers with some finite probability after overcoming them in order to simulate glide plane softening. Dislocations were assumed to move at sonic velocities between the short-range barriers. In other words, the limiting velocity of the dislocation in the absence of any barriers to dislocation motion was assumed to be the velocity of sound. In these calculations, the lead dislocations were observed to move slowly and as soon as a trailing dislocation in the slip band reached sonic velocities, the source generated a large number of dislocations. A dynamic pile-up was formed and the whole pile-up was noted to move at sonic velocities in an athermal fashion. In our case the trailing dislocations in the slip band will move at some finite velocity which is probably much below the speed of sound. The whole slip band propagates in a thermally activated fashion. In summary, the above discussion suggests that the assumption of the slip band moving at a constant velocity is very reasonable.

The second major assumption that was made was that only the leading dislocation is moving slower than a dislocation away from the tip. Rest of the dislocations forming the slip band were assumed to move at some constant velocity. Clément et al[17] have
shown, based on their in-situ deformation studies in a Ni-30wt% Cr alloy, that the velocity distribution of dislocations in a dynamic pile-up can be complex. Calculations of dynamic pile-up formation by Arsenault showed that the velocity of the first few dislocations trailing the lead dislocation are in fact lower than that of the lead dislocation. But once a trailing dislocation reached the terminal velocity (which was taken to be the speed of sound in his calculations), a dynamic pile-up was formed and the band moved at a constant velocity. Detailed calculations of dislocation velocity distributions in a material exhibiting glide plane softening and with dislocations moving relatively slowly have not been performed by anyone. Our assumption, although simple and ignores some of the details, is reasonable when one considers the propagation of the entire slip band.

Finally, it is apparent that our velocity based arguments are complimentary to the arguments used for evaluating $\tau_{\text{SRO}}$ in FCC-SRO alloys from post-deformation pile-up structures. In principle both approaches should give the same results for a given $\gamma_{\text{SRO}}$. But, the success of our approach is dependent on capturing the dislocation positions in the pile-up when it was mobile. This will be difficult to achieve in the FCC-SRO alloys for the following reasons. The applied stress is lower than $\tau_{\text{SRO}}$ in the case of FCC-SRO alloys. Further, the lattice friction $\tau_{\text{PEIERLS}}$ is very low in these alloys. Both factors conspire to alter the dislocation positions during unloading, once the slip band becomes immobile. In addition, a dislocation pile-up can become immobile due to fluctuations in solute concentrations. In such a case one cannot use the velocity arguments. Since, $\tau_{\text{PEIERLS}} \approx 0$ for these alloys, it does allow the evaluation of both $\tau_{\text{SRO}}$ and $\tau_{\text{SS}}$ from post deformation pile-ups. As noted by Jouaid et al[19], these evaluations will give only a lower bound for $\tau_{\text{SRO}}$ and one needs to perform in-situ deformation experiments to determine $\tau_{\text{SRO}}$ accurately. In conclusion, the velocity arguments proposed by us will
work if one can be sure that the pile-ups were in fact mobile and the dislocation positions have not been altered from their mobile configuration during unloading.

In addition to the sources of errors discussed above, there is one additional factor that needs to be considered when evaluating the magnitude of $\gamma_{\text{sro}}$. A key assumption in our arguments is that the whole slip band experiences the same stress. There will be errors in the estimates of $\gamma_{\text{sro}}$ if there is a stress gradient across the slip band. Stress gradients will exist in a real crystal due to the presence of other dislocations and their non-uniform distribution in a grain. These effects will be important for longer slip bands with hundreds of dislocations and will lead to complex dynamics within a slip band as observed by Clément et al.[17] in their in-situ studies. In the case of Ti-6Al, we have shown that the applied stress is higher than $\tau_{\text{sro}}$, implying that the pile-ups observed in post-deformed microstructures were in fact mobile under stress. Since $\tau_{\text{peierls}}$ is very high for the screw dislocations, it is reasonable to expect that the dislocation positions have not been altered significantly from their mobile configurations upon unloading and subsequent thin foil preparation. This is further supported by the lack of backflow upon complete unloading. The estimates of $\gamma_{\text{sro}}$ were conducted on pile-ups which are small with only few dislocations in them. Therefore, these estimates should be reasonably accurate.

6.5 Modeling of primary transient based on dislocation dynamics

In the previous sections, many different features of the deformation behavior of a Ti-6Al alloy was discussed in detail. There were major differences in dislocation structures between samples that were IWQ and all the other samples. In this section, the primary
transient of the samples that were heat treated to modify the SRO state will be discussed first and IWQ samples will be discussed at the end.

Figure 6.42 shows schematically the sequential operation of a dislocation source and the subsequent motion of dislocations and formation of screw slip bands, based on the discussions presented before. The dislocation at the source expands, elongated along the screw orientation. Once the separation distance between the screw dipole exceeds the critical separation ($L_c$) the edge segments run out to the other end of the grain. As further dislocations are emitted by the source, screw slip bands are formed, which move and pile-up against the grain boundary. The schematic also shows that the dislocations tend to orient themselves along the grain boundary, as observed in deformed microstructures. But the dislocations slowly change their orientation and eventually, far away from the boundary, they are in the screw orientation.

One of the features of the observed room temperature creep in these alloys is the lack of backflow upon a complete load drop. This is shown in figure 6.43. This is intriguing because the deformation microstructure consists of large number of pile-ups. It was shown in figure 6.30, and as discussed above that the dislocations are in mixed orientation near grain boundaries and piled-up against the grain boundary. The reason for the lack of backflow is because of the large Peierls stress on the screw dislocations. The dislocations that are in the screw orientation in the pile-up could be locked the moment the sample is unloaded. This would prevent the piled-up dislocations at the grain boundary from altering their positions by backflow. Hence, there is no significant backflow in these alloys.
Slip line evolution studies provided direct insight into the primary transient as discussed in section 6.3.5. Based on this we learnt that the initial transients are determined by the deformation dynamics of the most favorably oriented grains. Following this, the dynamics of slip communication and deformation dynamics of less favorably oriented grains dominate the primary transient shape. The final transient seems to be controlled by interaction of parallel slip bands and slip band intersections, as many grains both favorably oriented and unfavorably oriented are participating in the deformation.

Among the samples that have been heat treated to alter the SRO state, three kinds of transients have been observed. IWQ-age samples exhibit three regimes of exhaustion rates, AC samples exhibit only two regimes of exhaustion. Both SQ-400C-5h and SQ-600C-24h-400C-5h samples exhibit complex primary transients. There is an initial "incubation" period, followed by a very short inverse primary, then a normal primary and an extended inverse primary, which is followed by final normal primary transient (see figure 6.9).

Upon loading, sources in the most favorably oriented grains are activated. The edge segments will run out to the other end of the grain and get piled-up. With time screw pile-ups will be established which will move laterally away from the source. The initial strains and strain rates will be determined by these dislocation processes. Slip nucleation in the neighboring grains (secondary grains) will occur when sufficient stress concentration has been built up at the grain boundaries. Based on our discussion above this will occur initially at those locations near the grain boundary where edge pile-ups are formed. Even though the edge segments move much faster than the screw segments, the time needed for the stress build up will still be determined by the dynamics of the screw
segments. This is because of the way the source operates. In order to generate a new
dislocation at the source, the screw segments that were emitted before will have to move
away a sufficient distance from the source. Once emitted, the edge segment will run to
the grain boundary and pile-up there (see figure 6.42). Hence, there will be a finite time
required for the second "yielding" process. Upon this second "yielding" the dislocations
in the less favorably oriented grains will move at a lower rate, because they will be
experiencing a lower applied stress. Similar, source dynamics and dislocation processes
will occur in these secondary grains, as discussed above. One would expect the dynamics
to be slower in the secondary grains. With time, slip will be communicated to the entire
sample and communication back to the primary grains could occur after long times. At
this point most of the grains will be deforming in the sample and the primary transient
will be determined by a combination of processes occurring in many grains.

One can envision the entire primary transient to be composed of three transients. The
initial transient is determined by the deformation dynamics of the favorably oriented
grains. There will be a second transient associated with the secondary grains, which will
be superimposed on the overall transient after the time required for stress build up for the
second yielding. Once slip has been communicated to all the grains, there will be an
overall transient which is determined by deformation dynamics of many grains deforming
simultaneously.

When the deformation rate of the initially yielded grains are high, the time required for
slip nucleation in the neighboring grains will be less. Since the unfavorably oriented
grains deform at a lower rate, their transient will be masked by the deformation transient
of the favorably oriented grains. It will be possible to distinguish all these different
transients, if these processes occurred sequentially, i.e. if the initially yielded grains stop deforming, once the second set of grains start deforming. This will not happen in a real material. All these processes occur simultaneously. But if the favorably oriented grains deform at a lower rate, i.e. at a rate comparable to the secondary grains, these transients will be noticeable.

In the SQ-400C-5h sample and the SQ-600C-24h-400C-5h samples, the initial strains and strain rates are very low. From the dislocation processes illustrated in figure 6.43, it follows that the instantaneous strain will be determined by the motion of the edge segments of the "hair-pin" structures. The strain rates associated with this process should be relatively high and hence, it is treated as an instantaneous strain. Further straining and hence the strain rates will be determined by the lateral motion of screws and the establishment "mobile" screw pile-ups. Due to the presence of SRO, this process could be very slow. Emission of additional dislocations after the initial "hair-pin" structures are formed, could be a slow process. This is because, the source will experience a back stress due to the presence of the screws. Therefore, further emission of dislocations will not occur until the screws move apart a distance such that the dislocation source experiences an effective positive stress. This process implies that the screw dipole separation distance will have to continually increase with the number of dislocations in these nascent slip bands in order for the source to continue to emit dislocations. All the above suggests that source dynamics will be slow until there are sufficient number of dislocations in a nascent slip band to cause enough stress amplification on the lead dislocation to increase the velocity of the band.
The initial “incubation” observed in these two samples, where the strain rates are very slow for tens of seconds, is attributed to the time dependence of the establishment of “mobile” screw pile-ups. And the initial inverse primary probably occurs when sufficient “mobile” screw pile-ups are established and this is coupled with the increased velocity of these nascent slip bands when sufficient number of dislocations are added to them. Since the strains and strain rates are small in the first regime, we observe the primary associated with the favorably oriented grains. The second inverse primary is probably associated with slip nucleation in the neighboring grains. Since the strain rates are low in the initial regime, nucleation of slip in the grains neighboring the favorably oriented grains leads to a noticeable acceleration in creep rates.

As noted in section 6.2, such “incubation” periods have also been observed in AC and SQ-600C-24h samples. Further, the “incubation” period in the SQ-400C-5h (≈10.5s) is lower than that in the SQ-600C-24h-400C-5h sample (≈40s). The first reason one can think of is that the SRO state is stronger in the SQ-600C-24h-400C-5h sample. But the \( \gamma_{SRO}/\tau_{SRO} \) data presented for the AC and SQ-600C-24h-400C-5h samples suggests that the differences in the magnitude of \( \gamma_{SRO} \) between all the different heat treatments are probably very subtle. This is also indicated by the neutron diffraction data presented in chapter 4, where there are no obvious differences in the neutron scattering data between the different ageing treatments. Considering the screw pile-up formation process described above, longer “incubation” period implies that either the number of sources that are active in the favorably oriented grains is lower or that the dynamics of the mobile screw pile-up formation is slower or both. Let us consider the first possibility. Detailed dislocation characterization of creep deformed SQ-600C-24h-400C-5h sample indicated that the lead four or six dislocations in a slip band are paired in this sample. Also the edge
pairs of the “hair-pin” structures are strongly paired. Even though estimates of $\gamma_{SRO}^2$ for both AC and SQ-600C-24h-400C-5h samples appear to be very similar, the strong pairing of the lead edge segments in latter, indicates that $\gamma_{SRO}^1$ is significantly higher for the SQ-600C-24h-400C-5h sample. During the activation of a source, when the first dislocation is emitted by the source, the creation of $\gamma_{SRO}^1$ opposes dislocation emission. Hence, it is reasonable to expect the number sources active in the favorably oriented grains to decrease in the SQ-600C-24h-400C-5h sample.

Let consider hypothetically two samples with low and high $\gamma_{SRO}$ values. During the formation of “mobile” screw pile-ups the trailing dislocations will pile-up against the leading dislocation(s) in order for the band to move at a constant velocity. For the two cases considered, dislocations in a slip band will be piled-up stronger in the case of the sample with higher $\gamma_{SRO}$. This also implies that there will be a larger back stress on the last dislocation, thus, decreasing the effective stress on that dislocation. Such a process can significantly affect the dynamics of the formation of “mobile” pile-ups and could significantly slow down this process. Both reduction in sources and slower dynamics of formation of “mobile” screw pile-ups can act together and cause the observed differences. Even though the difference in $\gamma_{SRO}$ should be relatively small, subtle differences in the dynamics can collectively cause significant differences in the macroscopic response. These arguments should be valid whenever sources are activated during the deformation at various stages of the primary transient. The fact that the second inverse primary occurs at an earlier time for the SQ-400C-5h sample compared to SQ-600C-24h-400C-5h sample and that the total strains accumulated by the SQ-600C-24h-400C-5h sample is lower is consistent with these arguments. Because when slip is communicated to neighboring grains, one will need a larger stress amplification for
source activation and the dynamics of these sources will be slower, leading to longer times before the second inverse primary occurs. Finally, reduction in the number of activated sources over many grains will lead to lower total accumulated strains as observed in the SQ-600C-24h-400C-5h sample.

In contrast to the SQ-400C-5h and SQ-600C-24h-400C-5h samples discussed above, the AC samples do not exhibit any second inverse primary. Some AC samples did show the initial "incubation" period. Otherwise, the AC samples exhibit only regimes of exhaustion. An initial regime of low exhaustion rate and a final regime similar to other samples. The absence of a regime with a high exhaustion rate initially is consistent with the low initial strains and strain rates observed in these samples. Deformation rate in the favorably oriented grains are probably comparable to those in the less favorably oriented grains, but the initial rates are still large enough that the slip nucleation in the neighboring grains does not cause an observable increase in strain rate. This causes the exhaustion rate to remain low for a long duration. Figure 6.10 shows the variation of creep rate with time for an AC sample, which supports these arguments.

IWQ-age samples exhibit three regimes of different exhaustion rates. There is an initial regime of high exhaustion rate followed by a regime of low exhaustion rate and then a final regime of higher exhaustion rate again. The main difference between these samples and those discussed above is the occurrence of this initial regime of high exhaustion rate. Also, no "incubation" period was observed. Instead, the initial creep strains and strain rates are rather high (see table 6.2). This suggests that the source dynamics in terms of formation of screw pile-ups is relatively fast. Considering the initial response of the SQ-600C-24h sample it is reasonable to expect significant initial strains from "hair-pin" like
structures. But, that sample exhibited an initial “incubation” period. This is not observed in IWQ-age samples. Significantly different source dynamics implies that $\gamma_{\text{SRO}}$ is rather low but, it is not reasonable to expect significantly lower $\gamma_{\text{SRO}}$ in these samples from the neutron diffraction results. An alternative possibility is that stronger and weaker regions of SRO were generated during these heat treatments. From the neutron diffraction studies of IWQ samples, we know that there is some weak SRO present in the as quenched state. When samples with such an initial state are subsequently aged, the SRO present at the start could bias the subsequent ageing treatment, leading to stronger and weaker regions of SRO. Such a possibility is not unreasonable. Figure 6.7 shows the creep response of three samples, SQ-600C-24h, SQ-400C-5h, SQ-600C-24h-400C-5h. Among the three samples, the SQ-600C-24h-400C-5h sample is the strongest. This clearly shows that the SRO state obtained by modifying the SRO state that was present after the SQ-600C-24h treatment by a lower temperature age is very different from the SRO state obtained from direct ageing at the lower temperature. Similar behavior is observed in the AC samples as well. The initial transient of the samples that were subsequently aged after the AC treatment is very different from the as AC samples (see figure 6.5). Recently, Liew[76] studied decomposition of single phase $\alpha$ into $\alpha$ and $\alpha_2$ in a Ti-15at% Al alloys by computer simulations. She noted that in their simulations certain atomic sites designated as “ordered sites” acted as nuclei for the formation of an ordered domain. Ordered sites are those atomic sites which have the correct nearest-neighbor arrangement with respect to the $\alpha_2$ structure, even in a random arrangement of atoms. Such observations indicate that it is reasonable to expect the prior SRO structure to bias the subsequent ageing. Finally, measurements of $\gamma_{\text{SRO}}$ in a SQ-600C-24h-400C-5h sample from dislocation pile-ups showed that there is some non-uniformity in the SRO structure. All these factors suggest that in the case of IWQ-age samples, deformation in the weaker regions
contribute significantly to the initial transient. Influence of such modulations in SRO strength on the deformation behavior can be cleanly observed only during the initial transient. Effect of such modulations on the deformation behavior of secondary grains should be less. Since the deformation of favorably oriented grains will determine the behavior of the secondary grains, the overall transient for these samples are different.

In summary, various dislocation processes have been identified that could explain the observed primary transients after different ageing treatments. There are significant differences in the creep response, mainly in the initial transients of these samples. The long time transients are very similar for all the samples. During the final transient most of the grains in the sample are deforming and significant strains have been accumulated. At this stage, interactions between parallel slip bands and slip band intersections probably determine the creep transient therefore, is very similar in all the samples. The total strain accumulation is also very similar for all the samples, except for the SQ-600C-24h-400C-5h sample which accumulated significantly less strains even after very long times. As discussed above, it is possible that number of active sources did decrease in this sample and caused smaller accumulation of creep strains.

So far all the discussion have been focussed on the primary transients of the aged samples. A brief discussion of the primary transient of IWQ samples will made now. It was shown that the deformation is homogeneous in the case of IWQ samples(section 6.3.2). There was also evidence for lot of cross-slip. Regarding the primary creep transient of these samples, they showed three regimes of exhaustion similar to IWQ-age samples. Initial creep strains and rates are relatively high (except for IWQ-1) in these samples. Dislocation processes determining the creep transient are expected to be
different here. Source dynamics are probably not very important because slip can spread from initial few sources by cross-slip. The mechanism for communication of slip to grains neighboring the favorably oriented grains will also be different. As noted in section 6.3.2, slip becomes very diffuse near the grain boundaries. Hence, strong interaction of planar slip bands with grain boundaries is not present in these samples. The slip accommodation process involves compatibility of the grain as a whole and not accommodation of local incompatibilities. Thus, the regime of lower exhaustion rate is caused by slip accommodation in grains neighboring the favorably oriented grains. But the microscopic processes are very different from the aged samples. A consequence of the deformation characteristics of IWQ samples is that texture can significantly influence the overall creep behavior. Because, deformation occurs homogeneously over the whole grain and local incompatibilities are not present. As noted in section 6.1, there is a significant spread in the total creep strains between the five IWQ samples. Differences in the texture of the samples could explain the spread in the creep strains among the IWQ samples.

6.6 Discussion of room temperature creep in titanium alloys

Room temperature creep has been observed to occur in titanium alloys at stresses as low as 25% of the yield stress[77] and for times as long as 27 years[78]. Most of these studies have been conducted on two phase α/β titanium alloys. Hence, a direct comparison of the results from this study to those in the literature cannot be made. But an attempt will be made to rationalize some of the results in the literature based on the understanding gained from our work. A number of reasons have been proposed to explain the phenomenon of room temperature creep, both microstructure based and phenomenological. We will
discuss some of the microstructural arguments first and then discuss briefly the phenomenological arguments, which have been discussed in detail in chapter 5.

Thompson and Odegard[79] studied room temperature creep in a single phase alloy Ti-5Al-2.5Sn. Three different microstructures were studied, as-received condition, ρ-forged (forged at 954°C and air cooled) and β-forged (forged at 1052°C and air cooled). The as-received microstructure consisted of equiaxed grains of 50μm in size, the β-forged alloy had a martensitic microstructure and the ρ-forged structure consisted of equiaxed grains of 3μm. Among the three conditions, the as-received condition was the least creep resistant and the ρ-forged condition was the most. The β-forged sample had intermediate creep response. The differences between the three conditions were only significant when crept at 60% of the yield stress. At a stress level of 90% of the yield stress there was no significant difference between the three microstructures. Thompson and Odegard[79] proposed that the differences in creep response was due to differences in the nature of dislocation sources present in the three microstructures. They proposed that upon loading, easiest sources are activated first and they are exhausted with time leading to the activation of more difficult sources and this leads to a continuous decrease in creep strain with time. Based on this argument, it was suggested that dislocation sources were difficult to operate in the ρ-forged material causing it to be more creep resistant. In addition, they also proposed that pre-straining the material will decrease the creep strains, because such a process will exhaust the "easy" sources.

Odegard and Thompson[80], in their studies on Ti-6Al-4V, applied the dislocation source approach and tested the alloy at a lower stress level after pre-straining at a higher stress level. They observed that the amount of pre-straining required to retard creep, increased
with increase in the applied stress level. They also noted that at the higher stress levels pre-straining in fact lead to an increased creep rate. Odegard and Thompson[80] also tested samples after chemical polishing the sample surface to remove the damage layer due to machining. These samples were tested at 60% of the yield stress and they showed that chemical polishing reduced the accumulated creep strain, when compared to a sample that was not polished. This result was also thought to provide evidence that dislocations sources controlled the room temperature creep of titanium alloys. Because, dislocation sources activated by the machining process were removed by chemical polishing. Such an argument will be valid only at lower stress levels, because the contribution of strains from the surface grains could be a significant fraction of the total strain. But if one were to test the alloy at higher stresses, the effect of such a process will be minimal. Indeed, Odegard and Thompson[80] stated that there was no significant difference between the polished and unpolished samples when they were tested at 70% of the yield stress.

Cottrell[81] has proposed a low temperature creep model based on exhaustion of dislocation sources. This model led to a logarithmic creep law. But in titanium alloys a power law in time is observed (equation 5.1), not a logarithmic creep law. In polycrystals, grain boundaries are important sites for dislocation sources[32]. If one takes this view, then the α-forged sample, which had a grain size of 3μm, will have a greater density of sources and hence, should creep at a higher rate. Further, it has been shown that extrinsic grain boundary dislocations, those dislocations which were generated by slip and accommodated at the grain boundaries, can assist grain boundary dislocation sources in generating dislocations at a lower stress[82]. In the work of Thompson and Odegard, the α-forged material was only partially recrystallized. Hence, this sample should have a
significant number of extrinsic grain boundary dislocations, leading to easier source activation. Both factors would suggest that α-forged material should be the least creep resistant, if dislocation sources controlled the creep deformation.

Detailed microstructural studies were conducted by Chen et al[83] and Miller et al[84] on a Ti-6Al-2Nb-1Ta-0.8Mo (Ti-6211) alloy. They suggested that room temperature creep in titanium alloys is controlled by the slip length of the microstructure. Slip length was defined as the distance between barriers to slip. Two samples, one with a widmanstätten colony microstructure and another with a basketweave microstructure tested by them, among many different samples, will be discussed here. The sample with the colony microstructure accumulated creep strains about twice as high as the sample with basketweave structure. It was argued that there is a Burgers orientation relationship between the α and β laths in a colony microstructure. Therefore, the β laths do not offer any resistance to slip in this case. Slip length for such a microstructure was assumed to be the colony size. In the case of basketweave microstructure, it was stated slip length is small in this condition because the colony size was much smaller and there were several α/β interfaces lacking Burgers orientation relationship. Since the size of a colony is much larger, it was concluded that the larger slip length is reason for poorer creep resistance of the colony microstructure. Recently, it has been shown by Suri et al[85] that slip transmission across the β laths is not an easy process. It depends on the specific slip system that is active in the α phase and between the different prism slip systems, such a transmission process is highly anisotropic in nature. Hence, it is not reasonable to arbitrarily consider the colony boundary to be the slip length for widmanstätten microstructures. Further, the slip length argument is similar to a Hall-Petch argument for creep. If one looks at the 0.2% yield strength between the two microstructures, it is
760MPa for the colony microstructure (their label, W1) and is 751MPa for the basketweave microstructure (their label, W8)\[83\]. The large decrease in the slip length does not seem to increase the yield strength of the basketweave microstructure as one might expect. In fact it has a slightly lower yield strength. Finally, it was shown by Bowden and Starke\[86\], that after tensile deformation of 4% plastic strain, slip lines flowed across many colonies of the basketweave microstructure in the same alloy studied by Chen et al and Miller et al. All the above show that the slip length argument is strong but is not complete.

From our studies, we know that in the $\alpha$ phase, slip is planar due to the presence of SRO. This leads to the formation of pile-ups in the microstructure. Therefore, interaction of these pile-ups with both grain boundaries and interphase boundaries are important during plastic deformation. Such planar slip was observed in the $\alpha$ phase in all the works that were discussed above. Since the deformation microstructure consists of pile-ups, slip lengths are important for understanding the microstructural effects on the room temperature creep behavior. Under creep conditions, it is reasonable to expect the weakest link in the microstructure to control the overall deformation. In the case of colony microstructure, since such a microstructure is produced by slow cooling, we can expect modulations in the strength of SRO present in the $\alpha$ phase. Whereas in the case of the basketweave microstructure, such modulations will be less because the samples are air cooled to produce the microstructure. (Note: In the work of Chen et al, in their condition W8, samples were aged at 950°C for 4 hours and air cooled after the initial treatment to produce the basketweave microstructure. Based on our studies, even after this additional ageing the SRO state should be similar to an air cool, because the samples were aged well above the $\alpha/\alpha+\alpha_2$ transus for the composition of the $\alpha$ phase in Ti-6211).
Since deformation is controlled by dislocations in pile-ups, smaller slip length should lead to lower stress concentration at boundaries. Therefore, will lead to lesser slip communication to neighboring grains. Even though the deformation of colony structure will be anisotropic, there are favorable orientations available where long slip bands can be established leading to significant creep strains. In the case of basketweave microstructures, such long slip lengths cannot be established due to smaller colony sizes. These differences are less apparent during constant strain rate deformation because under those conditions the material is forced to deform at a constant rate. Hence, preferential selection of weak-links in the microstructure will not occur. But, these differences will be magnified under creep conditions. Such interaction between pile-ups and grain boundaries also could explain the results of Thompson and Odegard[80] on Ti-5Al-2.5Sn. In their studies, α-forged material had a much lower grain size (3μm), as received material had the largest grain size (50μm) and the β-forged material had intermediate sizes (martensite plate width of 5μm). Smaller grain sizes provide smaller lengths for pile-up formation, leading to lower stress concentrations at grain boundaries. Smaller stress concentrations at grain boundaries will lead to lesser slip communication and the alloy will accumulate smaller creep strains. Such interactions will be exaggerated at lower stress levels, where the length of the pile-up is important for slip transfer. At higher stress levels slip can be transferred with shorter pile-ups, because there will be greater dislocation density in the pile-ups and greater stress concentrations. From these arguments, the creep response will not be very different between the three microstructures at higher stress levels. This is consistent with the observations of Thompson and Odegard.
From our studies, we know that in the presence of SRO, pre-straining will not be beneficial. Because, pre-straining would establish dislocation pile-ups by activation of sources which would not otherwise operate at the lower stress level. Pre-straining could make subsequent deformation easier and in fact would not exhaust sources. This is consistent with the observation of Odegard and Thompson[80] that there was creep acceleration at the higher stress levels due to pre-straining. In general, pre-straining will not be beneficial after conventional heat treatments, because an SRO state will be produced in the $\alpha$ phase. Pre-straining will be beneficial in samples which have been IWQ to minimize SRO. In this case, the deformation was observed to be homogenous and such pre-straining could help reduce room temperature creep.

Hatch et al[87] proposed that high strain rate sensitivity of titanium alloys, after certain heat treatments, as the reason for room temperature creep. They observed that when titanium alloys(Ti-6Al-4V, Ti-6Al-6V-2Sn and Ti-4Al-3Mo-1V) were solution treated high in the $\alpha$ phase field and then subsequently aged around 500°C for 6h, they exhibited significant creep at stresses lower than their 0.01% yield strength. This lead to the suggestion that these alloys were more strain rate sensitive after such a treatment. This argument was extended by Odegard and Thompson[80], who suggested that creep testing is equivalent to performing constant strain rate tests at very low strain rates. This implies that the creep stress levels can be higher than the yield stress at those low strain rates. Whereas, Wapniarsky et al[88] suggested that the cause for room temperature creep in Ti-6Al-4V was the poor work-hardening characteristics of this alloy.

In summary, results presented in this work and the results from the literature indicate that there are two main features pertaining to the low temperature creep of titanium alloys. a)
That creep occurs at stresses well below the macroscopic yield stress and b) this creep process occurs for very long times. In chapter 5, it was shown that the strain rate sensitivity of titanium alloys is not abnormally high. Further, the initial creep rates can be very high and comparable to strain rates in constant strain rate tests after some heat treatments. It was stated in chapter 5 that the unusual room temperature creep of titanium alloys can be rationalized as due to a combination of moderate strain rate sensitivity, $m$ and abnormally low work-hardening exponent, $n$. Mechanistically it can be understood in the following way. Strain rate sensitivity of titanium alloys, is presumably due to the thermally activated motion of screw dislocations, which control the plastic deformation in titanium alloys. We have shown that, eventhough there will be SRO strengthening in these alloys after conventional heat treatments, the applied stresses are in general well above $\tau_{SRO}$ and the edge/mixed segments could be mobile upto fairly low stresses. And given enough time the screw dislocations will move at stresses lower than the macroscopic yield stress. Therefore, dislocations will be mobile at stresses significantly lower than the macroscopic yield strengths. In addition, work-hardening characteristics of titanium alloys are very poor. Combination of these factors seem to cause extensive creep of titanium alloys at stresses lower than macroscopic yield strengths, which can persist for very long times.

6.7 Summary

(a) Effect of short-range order on room temperature creep behavior was studied in a Ti-6Al alloy. These results showed that the SRO state indeed dramatically affects the creep behavior. Significant differences in the initial transient was observed between the different aged samples and the long time behavior was more or less similar. Three kinds of primary transients were observed in the aged samples. IWQ-age samples exhibited
three regimes of different exhaustion rates. An initial regime of high exhaustion rate, a second regime of low exhaustion rate and a final, long time regime of relatively higher exhaustion rates. AC samples and SQ-600C-24h samples exhibited only two regimes of exhaustion. An initial regime of low exhaustion followed by a regime of higher exhaustion rates. Whereas, SQ-400C-5h, AC-400C-1h and SQ-600C-24h-400C-5h samples exhibited very complex primary transients. Upon loading, there was an initial “incubation” period of very low creep rates($\approx10^{-9}$ to $10^{-8}$ s$^{-1}$). The duration of this “incubation” period was different for each sample and was usually of the order 10s. The SQ-600C-24h-400C-5h sample exhibited an “incubation” period of 40s. (Such incubation periods were observed in AC and SQ-600C-24h samples as well). After this initial regime, there was a regime of inverse primary with increasing creep rates for a few seconds. This was followed by a normal primary and a second inverse primary which lasted for thousands of seconds. Finally, there was a normal primary which was similar to other samples. The SQ-600C-24h-400C-5h treatment produced an SRO state that provided significant creep strengthening.

(b) Presence of SRO was shown to promote planar slip in these alloys. Pairing of lead dislocations in the slip bands were observed. When samples were ice water quenched from high in the $\alpha$ phase field, it was shown that the SRO state was weakened and homogenous deformation was promoted. Slip bands which appeared to be planar in the grain interiors were observed to become diffuse due to extensive cross-slip as the dislocations move towards the grain boundary. Similar spread of slip from the initial set of slip planes was observed during in-situ deformation in a HVEM. IWQ treatment could prove very beneficial for commercial two phase titanium alloys, even more than that for the single phase alloy studied here.
(c) Slip line evolution studies provide some insight into the deviation from power law form observed at early times and strains. It is suggested that the initial, more rapid exhaustion is due to interaction of the few existing slip bands with grain boundaries. The regime where the exhaustion rate is less rapid may be associated with the generation of new slip bands near grain boundaries in grains for which deformation was not initially apparent. These newer slip bands propagate very slowly into the grains, causing a less rapid exhaustion. At long times, possible interactions between slip bands in parallel planes and interactions between intersecting slip bands have been noted, leading to higher exhaustion rates and the reduced time exponents observed at long times.

(d) From the observed dislocation structures, energy of the diffuse APB(γ_{SRO}) was estimated. Two approaches were used, one was based on the observed "hair-pin" like structures and the other was from dislocation pile-ups. In comparison to the studies on FCC-SRO alloys, we have suggested an alternative, dislocation velocity based arguments to rationalize the use of dislocation pile-up interaction stresses to estimate γ_{SRO}. Estimated γ_{SRO} values from both approaches are very similar and are comparable to those estimated in the FCC-SRO systems. These have been estimated to be between 17 mJ/m² to 54 mJ/m². Presently, these estimates have been done for slip bands on the prism plane. In titanium alloys, basal, prism and pyramidal planes are crystallographically different. Therefore, one would expect different energies on those planes compared to our estimates on prism plane.

(e) It was shown that in the Ti-6Al alloy studied there are no obvious dislocation sources present in the grain interiors. Dislocations are generated at grain boundaries in these
alloys, at least initially. Operation of such a grain boundary source was also shown. Post-mortem deformation microstructures consisted of \langle a \rangle type screw dislocation pile-ups. This is because the screw orientation is the slowest moving line orientation due to the non-planar core structure of \langle a \rangle type screw dislocations. A dislocation velocity law was derived for thermally activation motion of screw dislocations in the presence and absence of $\gamma_{SRO}$. It was shown that effect of $\gamma_{SRO}$ can be treated as a lowering of the stress assistance for kink pair formation due to the creation of diffuse APB. Therefore, dislocation velocity at a given stress is lowered in the presence of $\gamma_{SRO}$. It was argued that dynamics of the operation of dislocation sources in this alloy will be determined by the lateral motion of screw dislocations. Hence, presence of $\gamma_{SRO}$ could significantly affect the source dynamics. Combination of slow source dynamics and the number active dislocation sources is thought to cause the "incubation" period and the differences in the "incubation" period among the different samples. Observed primary transients were explained based on these dislocation processes and on the results from slip line evolution studies. It was suggested that the initial creep transients are determined by the deformation characteristics of favorably oriented grains. The regime of lower exhaustion is due to a combination of deformation in favorably oriented grains and the neighboring grains to which slip is communicated. The final transient is probably determined by interaction of parallel slip bands, intersection of slip bands and interaction of slip bands with grain boundaries because during this regime many grains are participating in the deformation.

\(f\) It was noted that this unusual room temperature creep in titanium alloys occurs at stresses well below the macroscopic yield strength and for very long times. In chapter 5 phenomenological analysis of room temperature creep showed one has to consider both strain hardening exponent \((n)\) and strain rate sensitivity \((m)\), of these alloys to rationalize
the room temperature creep behavior. The strain rate sensitivity of these alloys is presumably due to thermally activated motion of screw dislocations. Hence, one can expect the screw dislocations to move at stresses well below the macroscopic yield strength, given enough time. Our measurements of $\gamma_{SRO}$ and the detailed microstructural studies show that the edge/mixed segments can be mobile up to relatively low stresses. This is because the applied stresses have to be high in order to move the screw dislocations which control the deformation and therefore, it will usually be higher than $\tau_{SRO}$. The above discussion suggests that dislocations can be mobile up to relatively low stresses in titanium alloys. It was noted in chapter 5 that in titanium alloys the strain hardening exponent ($n$) is abnormally low. From comparison of work-hardening characteristics of FCC-SRO alloys with titanium alloys, it was shown that even though SRO tends to lower work-hardening rates, titanium alloys exhibit much poorer work-hardening characteristics than FCC-SRO alloys. Therefore, promotion of planar slip due to the presence of SRO cannot completely explain this behavior. Several microstructural features were identified which could explain the poor work-hardening characteristics of titanium alloys. It was noted that there is no multipole formation in titanium alloys during planar deformation. This is very different from the deformation microstructures of FCC-SRO alloys. Dislocations in the slip bands tend to align themselves along the plane of the grain boundary. Therefore, dislocations near grain boundaries are in mixed orientation, but they are in screw orientation in the grain interiors. Since, lattice friction stress is maximum at the screw orientation and minimum at the edge orientation, the stress distribution at the grain boundaries due to even a single slip band will be highly anisotropic. Coupled with the fact that there are no multipole formation it implies that grain boundaries would not be as strong barrier for slip propagation in titanium alloys. Planar slip minimizes interaction between dislocations. Main dislocation interactions are
interaction between dislocations in the slip band with dipole debris on the slip plane, elastic interaction between parallel slip bands and intersection between slip bands on different planes. First of these interactions do not provide significant work-hardening. Interaction between parallel bands do provide some work-hardening. Since, there is no tendency to form multipoles in these alloys, such interactions do not stop slip in many instances. It was observed that intersecting slip bands appear to pass through each other in many cases and retain planarity of slip. But in other cases slip band intersections do seem provide some work-hardening. A combination of all these factors seem to be responsible for poor work-hardening in titanium alloys. It is probably due to the low work-hardening rates that low temperature creep can persist for very long times in titanium alloys. Hence, it is this unique combination of moderate $m$ values and abnormally low $n$ values, cause low temperature creep in titanium alloys.
References


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Figure 6.1: Log-log plot of creep time versus creep strain of IWQ samples. Five different samples were tested. Samples 1, 4 and 2, 3 and 5 were heat treated in separate batches. The response of all the samples are similar. The creep rate is decreasing monotonically through out the test. All samples exhibit three regimes of different exhaustion rates. But, there is a significant spread in the total creep strains among the samples.

Figure 6.2: Creep response of samples aged at 500°C for various times. The response of the aged material is similar to the IWQ samples. There are only subtle differences between the different samples. The difference in the total strains are small except for the case of sample that was aged for 50h, which accumulated larger strains. (see text for details).
Figure 6.3: Log creep time versus log creep strain plot of IWQ samples which were aged at different temperatures for different times. All the samples showed three regimes of exhaustion.

Figure 6.4: Creep response of air cooled (AC0 samples. Two samples AC-1 and AC-3 showed an initial regime of very low creep rates ($\approx 10^4 s^{-1}$). Other than this difference, all samples show only two regimes of different exhaustion rates. An initial regime of low exhaustion and a final regime similar to other samples. Creep response of the all the samples are very similar at long times.
Figure 6.5: Creep response of AC-1 sample is compared with samples that were aged after the AC treatment. Overall creep behavior (except the initial transient) of AC-350C-5h sample is very similar to AC-1. Instantaneous creep strain increased during ageing at 400°C and is higher for longer ageing times. Sample aged for 1 h at 400°C showed an initial very low strain rate regime (≈10⁻⁹ s⁻¹) for a duration of ≈30s.

Figure 6.6: Comparison of creep response of samples aged at 650°C for 24h. One sample was IWQ and aged. Two samples were step quenched (SQ) to the ageing temperature. Creep behavior is very similar for all three samples.
Figure 6.7: Creep behavior of three samples with different SQ-age treatments are compared. All three samples showed an initial regime very low strain rates. Two of the samples, SQ-400C-5h and SQ-600C-24h-400C-5h exhibited complex transients with two inverse primary regimes. The SQ-600C-24h-400C-5h sample was the most creep resistant.

Figure 6.8: Comparison of the creep response of samples with many different treatments to alter the SRO state. Some features to note are, a) there are significant differences in the initial response and b) long time transient is similar for all samples.
Figure 6.9: Complex primary transients observed in SQ-400C-5h and SQ-650C-24h-400C-5h samples are illustrated here. Both creep strain versus creep rate, (a) and creep time versus creep rate, (b) plots show that there are two inverse primary regimes observed in these samples. One was observed shortly after loading and a longer inverse primary was observed after longer times. The second regime occurred at longer times and at smaller creep strains in the SQ-600C-24h-400C-5h sample.
Figure 6.10: Primary creep response of Ti-6Al is compared after various heat treatments to modify the SRO state. Three kinds of transients were observed and typical examples are shown here. The IWQ and the IWQ-age samples show three regimes of different exhaustion rates, an initial rapid exhaustion regime, a second regime of slower exhaustion and a final regime of higher exhaustion. In contrast, AC samples show only two regimes of exhaustion. Primary transient of SQ-600C-24h-400C-5h sample is very complex. After the initial "incubation" period, there is a short inverse primary. This is followed by a normal transient and then a second (longer) inverse primary, after that a final transient similar to the other samples.
Figure 6.11: In (a) three slip systems are shown, labelled A, B and C. When imaged with \( g = (10\bar{1}1) \), slip system B is invisible and the slip trace is parallel to the trace of \((0001)\), this is shown in (b). This indicates slip system B to be \( a/3[1\bar{2}10] \) \((0001)\). In (c) slip system A is invisible for \( g = (1\bar{1}01) \), indicating the burgers vector to be \( a/3(11\bar{2}0) \). The dislocations are imaged with \((0002)\) reflection in (d). Slip systems A and B are invisible confirming that they are \(<a>\) type slip vectors. But system \(<c>\) is visible when imaged with \((0002)\), indicating that they are \(<c+a>\) dislocations. Finally, slip system A is imaged with \( g = (10\bar{1}1) \) in (e). This was imaged in zone axis \([1\bar{2}3]\) showing the slip system is edge on and parallel to the trace of \((\bar{1}00)\). Hence, slip system A is identified as \( a/3(11\bar{2}0)(\bar{1}00) \). (continued)
Figure 6.11: continued.

(c) $ZA = [01\bar{1}1]$

(d) $ZA = [\bar{1}2\bar{1}0]$
Figure 6.12: $<c+a>$ activity near a grain boundary in an AC sample is shown here. In most of the samples that were analyzed $<c+a>$ activity was concentrated near grain boundaries. In many cases these dislocations were observed to propagate into the grain interiors (see figure 6.13).
Figure 6.13: Montage of a creep deformed grain in an AC sample is shown in (a) and (b). Planar slip is observed and the deformation is concentrated in long pile-ups of screw dislocations. Little dislocation activity is present between the slip bands. All the planar bands comprise of \(<a>\) dislocations, but there is \(<e+a>\) activity and these are labelled in the figure. The extrapolated location of the dislocation source is indicated as S in (b). (continued)
Figure 6.13: continued
Figure 6.14: Shows planar deformation in a SQ-600C-24h-400C-5h sample. Slip is concentrated in screw pile-ups and there is little activity outside these bands. The slip bands shown are \( a/3[2110](0110) \).

Figure 6.15: Tip of an array in a SQ-600C-24h-400C-5h sample. It can be seen that the lead four dislocations are paired, indicated as LP. This slip band is the same slip system as that is shown above.
Figure 6.16: Observation of homogeneous slip in an IWQ sample. The deformation appears to be planar at the start (left side), but becomes diffuse due to extensive cross-slip as they move towards the grain boundary to the right (not shown here). All the dislocations are \(<a>\) type and most of them lie on the basal plane. Similar observations have been made on the prism plane as well. Such behavior was observed in the IWQ sample during in-situ deformation in a HVEM as well.
Figure 6.17: Dislocation propagation in an IWQ sample during in-situ deformation in a HVEM. It shows that slip spreads laterally from a few slip planes as it propagates towards the grain boundary. This is similar to what was observed in post-deformed microstructures that was shown in figure 6.16.
Figure 6.18: Montage of an undeformed sample after an IWQ treatment. The grain interiors are dislocation free, indicating that grain boundaries have to act as dislocation sources at the start of deformation. There is one dislocation present inside a grain, which is circled.
Figure 6.19: Operation of a grain boundary source in a SQ600C-24h-400C-5h sample. The $<a>$ dislocations are visible and can be observed to come from the grain boundary in (a). In (b) $<a>$ dislocations are invisible when imaged with $g=(0002)$. Dislocations A and B which are $<c+a>$ dislocations are labeled for reference between the two images. The grain boundary is labeled as GB.
Figure 6.20: A grain boundary source operating during in-situ deformation in a HVEM. The source is oriented to produce dislocations in the screw orientation.

Figure 6.21: (a) Two bands of opposite sign (P and N) are observed to pass through without forming strong multipoles in a SQ-600C-24h-400C-5h sample. Although there is no multipole formation, the bands do experience some difficulty when passing each other. This is illustrated in (b), where two bands (A and B) are passing each other. (see text for details). (continued)
Figure 6.22: (a)-(c) schematically illustrates the formation of debris loop due to cross-slip events on the slip plane. (d) illustrates possible interaction between a trailing dislocation on the slip plane with dipole debris formed by a leading dislocation in the slip band. Labels 1 and 2 refer to the parallel slip planes the dislocation segments lie on.
Figure 6.23: Three parallel prism slip bands (A, B and C) with $a/3[\overline{2}110]$ dislocations in a SQ-600C-24h-400C-5h sample. The dislocations in the bands are moving from the right/bottom to left/top of the image. One can observe several debris loops (DL) left by the leading dislocations in the band (see band C). Interaction between trailing dislocations and the debris loops can be significant (see bands A and B). (see text for details).

Figure 6.24: Interaction between two prism bands $a/3[\overline{2}110](0\overline{1}10)$, band A and $a/3[\overline{1}2\overline{1}0]$ (10\overline{1}0), band B in a SQ-600C-24h-400C-5h sample. Dislocations in band B are invisible and the slip plane is edge on. Even though there is significant interaction between the two slip bands, they seem to pass through each other. It is also clear that the dislocations stay planar after such an interaction.
Figure 6.25: Complex interaction between slip bands of various slip systems in a SQ-600C-24h-400C-5h. Slip bands labeled A belong to $a/3[\overline{2}110](01\overline{1})$, band B is $a/3[\overline{2}110](01\overline{1})$, slip bands C belong to $a/3[\overline{T}2\overline{T}0](1\overline{0}\overline{1})$ and band D is $a/3[\overline{T}2\overline{T}0](000\overline{1})$. (see text for details).

Figure 6.26: Intersection between basal and prism slip bands, assuming that the basal band was established first, is shown schematically. It is clear that the dislocations in the prism band will intersect the basal band at many locations, this is shown in (a). After such an intersection process, the dislocations on the prism plane will be heavily jogged, as shown in (b). (see text for details).
Figure 6.27: Schematic showing intersection between two prism bands. Unlike the prism/basal intersection, here the dislocations will penetrate the intersecting band in a sequential fashion.

Figure 6.28: At the early stages of plastic deformation, it possible that when slip bands intersect, they bow around each other band without cutting. Such a process is shown above schematically.
Figure 6.29: Interaction of $<a>$ type slip bands with $<c+a>$ dislocations in an AC sample. Even though there is significant interaction, the dislocations in the slip band seems to pass through the $<c+a>$ dislocations.

Figure 6.30: Interaction between slip bands and grain boundaries is shown here. Slip bands in a SQ-600C-24h-400C-5h sample is shown in (a). One can observe that the dislocations in band A are in the mixed orientation(M) near the boundary, whereas they are in the screw orientation(S) away from the boundary. In (b), dislocations in a slip band in an AC sample are in the screw orientation even near the grain boundary. (continued)
Fig 6.31: Interaction of two slip systems, one prism and one basal is shown here. Note the passage of dislocations through the intersection with only minor difficulty. The prism system labeled A is (1100) +/-1/3[1120]. $b_1$ shown is the projected burger's vector of the dislocations in the prism plane. The basal slip system labeled B is (0001) +/-1/3[1210]. $b_2$ shown is the projected burger's vector of the dislocations in the basal plane.
Figure 6.32: Slip lines observed after creeping for 96 sec. Slip lines appear as both dark and bright lines at an angle. Slip traces are observed in grain A. The slip traces appear to hit the grain boundary GB. Some secondary slip can be observed in grain A. No slip lines were observed in grain B after 96 sec (not shown here). Horizontal dark lines are scratches made on sample surfaces to observe possible slip offsets.

Figure 6.33: Slip lines observed after creeping for 1000 sec. Notice the appearance of slip lines in grain B. The slip lines in grain B originate at the grain boundary. New slip lines and intensification of existing slip lines are observed in grain A. Additional secondary slip lines can be observed in grain A.
Figure 6.34: Slip lines observed after creeping for 9000 sec. Region indicated as I shows interaction between primary (P) and secondary (S) slip systems. See text for details.

Figure 6.35: Slip lines observed after creeping for $931 \times 10^3$ sec (258.5 hours). See text for details.
Figure 6.36: A screw pile-up in a SQ-600C-24h-400C-5h sample is shown in (a). From the observed inter-dislocation spacing, one can see that these are not classical pile-ups. Further, the interaction stresses between the dislocations oscillate and does not smoothly decrease to zero as one would expect for a classical pile-up, shown in (b). Both factors show that there is a large friction stress on the screw dislocations forming the pile-up.

Figure 6.37: The leading edge and screw segments forming the “hair-pin” structures (HP) in a SQ-600C-24h-400C-5h sample (a) and in an AC sample (b) are shown.
Figure 6.38: Schematic plot showing the change in $\gamma_{sro}$ with the number of dislocations ($n$) shearing the slip plane. See text for details.

Figure 6.39: Forces (excluding the applied stress) on the lead two edge dislocations forming the "hair-pin" configuration is shown above. When one considers the two dislocations together as a super-dislocation, such a dislocation experiences a drag due to $\gamma_2$ ($\gamma_{sro}$ after second dislocation). It is this quantity that has been evaluated from the "hair-pin" configurations.
Figure 6.40: Formation of a kink pair is shown schematically. The kinks were assumed to be abrupt edge kinks, which height, \( h \) and separation distance, \( l \).

Figure 6.41: Normalized dislocation velocity is plotted as a function of the applied stress. As \( \gamma_{\text{SRO}} \) increases, the velocity of the dislocation decreases when compared to a dislocation that is not creating any diffuse APB. Hence, in the screw pile-ups, the lead dislocation will be moving at a lower velocity when compared to a dislocation further inside the band. This is because the trailing dislocations will not experience a drag due to \( \gamma_{\text{SRO}} \) since they do not alter the diffuse APB. Such differences in velocity cause the dislocations to form a dynamic pile-up during deformation.
Figure 6.42: Based on the various microstructural observations, several dislocation processes were identified which are shown schematically above. Initially during the deformation, the edge segments bow out from the source. Once a critical separation between the screws is reached, the edge segments will run out to the other grain boundary. Dynamics of edge pile-up formation on the other side of the grain will still be controlled by the screw dislocations. This is because, further emission of dislocations can occur only when the screw dislocation move apart laterally a critical distance to minimize the back stress on the source. This critical distance will continue to increase with the emission each pair, as the back stress from the dislocations previously emitted by the source will continue to increase. Since the edge dislocations have more mobility than the screws, the microstructure will consists of only screw pile-ups. The dashed lines in (c) illustrate a cut during the preparation of foil for TEM, showing that such a process will leave only screw pile-ups in the microstructure. Finally, in (d), the dislocations have been shown to orient themselves along the grain boundary as per our observations.
Figure 6.43: Complete stress drop in a SQ-600C-24h-400C-5h sample. It shows that there is essentially no observable backflow. see also figure 8.1.
Table 6.1: Creep parameters of IWQ samples. $a_1$, $a_{II}$, $a_{III}$ are the creep time exponents in the three regimes of exhaustion.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Creep strain at t=1s</th>
<th>Instantaneous creep rate (s$^{-1}$)</th>
<th>Creep rate at t=950s</th>
<th>$a_1$</th>
<th>$a_{II}$</th>
<th>$a_{III}$</th>
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</thead>
<tbody>
<tr>
<td>IWQ-1</td>
<td>$1.5 \times 10^{-5}$</td>
<td>$4.9 \times 10^{-5}$</td>
<td>$3.3 \times 10^{-7}$</td>
<td>0.18</td>
<td>0.37</td>
<td>0.24</td>
</tr>
<tr>
<td>IWQ-2</td>
<td>$1.9 \times 10^{-4}$</td>
<td>$1 \times 10^{-4}$</td>
<td>$1.2 \times 10^{-6}$</td>
<td>0.36</td>
<td>0.43</td>
<td>0.22</td>
</tr>
<tr>
<td>IWQ-3</td>
<td>$1.8 \times 10^{-4}$</td>
<td>$8.6 \times 10^{-5}$</td>
<td>$3.8 \times 10^{-7}$</td>
<td>0.21</td>
<td>0.37</td>
<td>0.24</td>
</tr>
<tr>
<td>IWQ-4</td>
<td>$3.4 \times 10^{-4}$</td>
<td>$1 \times 10^{-4}$</td>
<td>$5.5 \times 10^{-7}$</td>
<td>0.18</td>
<td>0.32</td>
<td>0.25</td>
</tr>
<tr>
<td>IWQ-5</td>
<td>$2.4 \times 10^{-4}$</td>
<td>$9.5 \times 10^{-5}$</td>
<td>$6.9 \times 10^{-7}$</td>
<td>0.22</td>
<td>0.36</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Table 6.2: Creep parameters of IWQ-age samples. Samples were aged after the IWQ treatment to different times at various temperatures.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Creep strain at t=1s</th>
<th>Instantaneous creep rate (s$^{-1}$)</th>
<th>Creep rate at t=950s</th>
<th>$a_1$</th>
<th>$a_{II}$</th>
<th>$a_{III}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>IWQ-500C-0.5h</td>
<td>$2.4 \times 10^{-4}$</td>
<td>$2.9 \times 10^{-5}$</td>
<td>$6.4 \times 10^{-7}$</td>
<td>0.10</td>
<td>0.47</td>
<td>0.27</td>
</tr>
<tr>
<td>IWQ-500C-5h</td>
<td>$2.5 \times 10^{-4}$</td>
<td>$9.7 \times 10^{-5}$</td>
<td>$4.4 \times 10^{-7}$</td>
<td>0.17</td>
<td>0.33</td>
<td>0.28</td>
</tr>
<tr>
<td>IWQ-500C-10h</td>
<td>$1.3 \times 10^{-4}$</td>
<td>$6 \times 10^{-5}$</td>
<td>$6.7 \times 10^{-7}$</td>
<td>0.3</td>
<td>0.43</td>
<td>0.23</td>
</tr>
<tr>
<td>IWQ-500C-50h</td>
<td>$3.5 \times 10^{-4}$</td>
<td>$1.6 \times 10^{-4}$</td>
<td>$1.3 \times 10^{-6}$</td>
<td>0.21</td>
<td>0.43</td>
<td>0.21</td>
</tr>
<tr>
<td>IWQ-350C-100h</td>
<td>$1.1 \times 10^{-4}$</td>
<td>$4.9 \times 10^{-5}$</td>
<td>$2.4 \times 10^{-7}$</td>
<td>0.2</td>
<td>0.45</td>
<td>0.31</td>
</tr>
<tr>
<td>IWQ-600C-1h</td>
<td>$2.3 \times 10^{-4}$</td>
<td>$7.8 \times 10^{-5}$</td>
<td>$1.1 \times 10^{-7}$</td>
<td>0.18</td>
<td>0.46</td>
<td>0.25</td>
</tr>
</tbody>
</table>
Table 6.3: Creep parameters of samples that were deformed after an AC from 900°C. Samples AC-1 and AC-3 exhibited very low initial creep rates. All samples exhibited a regime of very low exhaustion rates initially and a regime of higher exhaustion rates at long times.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Creep strain at t=1s</th>
<th>Instantaneous creep rate (s⁻¹)</th>
<th>Creep rate at t=950s</th>
<th>α₁</th>
<th>α₂</th>
<th>α₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>IWQ-650C-24h</td>
<td>6x10⁻⁵</td>
<td>1.4x10⁻⁵</td>
<td>4.8x10⁻⁷</td>
<td>0.27</td>
<td>0.57</td>
<td>0.32</td>
</tr>
<tr>
<td>SQ-650C-24h-1</td>
<td>6.3x10⁻³</td>
<td>1.3x10⁻⁵</td>
<td>4.7x10⁻⁷</td>
<td>0.18</td>
<td>0.61</td>
<td>0.26</td>
</tr>
<tr>
<td>SQ-650C-24h-2</td>
<td>7.6x10⁻⁵</td>
<td>1.5x10⁻⁵</td>
<td>5.6x10⁻⁷</td>
<td>0.44</td>
<td>0.50</td>
<td>0.27</td>
</tr>
<tr>
<td>SQ-400C-5h</td>
<td>4.8x10⁻⁸</td>
<td>≈10⁻⁹-10⁻⁸(10s)</td>
<td>8.1x10⁻⁷</td>
<td>0.87</td>
<td>1.56</td>
<td>0.35</td>
</tr>
<tr>
<td>(t=4.9s)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SQ-600C-24h</td>
<td>2x10⁻⁴</td>
<td>≈10⁻⁹-10⁻⁸(11s)</td>
<td>5.7x10⁻⁷</td>
<td>-</td>
<td>0.56</td>
<td>0.26</td>
</tr>
<tr>
<td>(t=4.4s)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SQ-600C-24h-400C-5h</td>
<td>4.5x10⁻⁶</td>
<td>≈10⁻⁹-10⁻⁸(40s)</td>
<td>2.6x10⁻⁸</td>
<td>0.48</td>
<td>1.6</td>
<td>0.32</td>
</tr>
<tr>
<td>(t=18.4s)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 6.4: Creep parameters of samples that were step quenched (SQ) and aged.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Foil Normal</th>
<th>Operative slip systems</th>
<th>Schmid factor</th>
<th>Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>IWQ-3</td>
<td>[2313]</td>
<td>(0001) [1210]</td>
<td>0.49</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0110) [2110]</td>
<td>0.24</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(1011) [1110]</td>
<td>0.31</td>
<td>III</td>
</tr>
<tr>
<td>IWQ-1</td>
<td>[4841]</td>
<td>(0110) [2110]</td>
<td>0.43</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(1100) [1120]</td>
<td>0.43</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0001) [2110]</td>
<td>0.07</td>
<td>III</td>
</tr>
<tr>
<td>AC-1</td>
<td>[2750]</td>
<td>(1100) [1120]</td>
<td>0.5</td>
<td>I</td>
</tr>
<tr>
<td>AC-1</td>
<td>[4513]</td>
<td>(0110) [2110]</td>
<td>0.36</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0001) [1210]</td>
<td>0.42</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0110) [2110]</td>
<td>0.48</td>
<td>II</td>
</tr>
</tbody>
</table>

Table 6.5: Operative slip systems in IWQ and AC are listed here. Prevalence of the slip system in the analyzed grain is indicated by the ranking. Schmid factors were calculated assuming the foil normal to be the stress axis.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Foil Normal</th>
<th>Operative slip systems</th>
<th>Schmid factor</th>
<th>Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>IWQ-350C-100h</td>
<td>[3962]</td>
<td>(T100) [1120]</td>
<td>0.47</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0001) [T2T0]</td>
<td>0.22</td>
<td>II</td>
</tr>
<tr>
<td>IWQ-650C-24h</td>
<td>[T41516]</td>
<td>(01T0) [2110]</td>
<td>0.4</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(01T1) [2110]</td>
<td>0.49</td>
<td>III</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(10T0) [T2T0]</td>
<td>0.35</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0001) [T2T0]</td>
<td>0.3</td>
<td>II</td>
</tr>
</tbody>
</table>

Table 6.6: Operative slip systems in two IWQ and age samples are listed here. Ranking of the operative slip systems indicates the prevalence of the observed systems. The schmid factor was calculated assuming the foil normal to be the stress axis.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Foil Normal</th>
<th>Operative slip systems</th>
<th>Schmid factor</th>
<th>Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>SQ-600C-24h-400C-5h</td>
<td>[3611]</td>
<td>(01T0) [2110]</td>
<td>0.48</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(10T0) [T2T0]</td>
<td>0.33</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(1010) [1120]</td>
<td>0.12</td>
<td>III</td>
</tr>
<tr>
<td>SQ-600C-24h-400C-5h</td>
<td>[7923]</td>
<td>(01T0) [2110]</td>
<td>0.45</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(01T1) [2110]</td>
<td>0.5</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(10T0) [T2T0]</td>
<td>0.26</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0001) [T2T0]</td>
<td>0.29</td>
<td>II</td>
</tr>
</tbody>
</table>

Table 6.7: Operative slip systems in SQ and age samples along are listed here. Ranking of the operative slip systems indicates the prevalence of the observed systems. The schmid factor was calculated assuming the foil normal to be the stress axis.
<table>
<thead>
<tr>
<th>Alloy</th>
<th>Single glide work-hardening rate (MPa)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-0at%Cr</td>
<td>80</td>
<td>32</td>
</tr>
<tr>
<td>Ni-1.8at%Cr</td>
<td>59</td>
<td>32</td>
</tr>
<tr>
<td>Ni-4.36at%Cr</td>
<td>31</td>
<td>32</td>
</tr>
<tr>
<td>Ni-10.05at%Cr</td>
<td>24</td>
<td>32</td>
</tr>
<tr>
<td>Ni-13.6at%Cr</td>
<td>20</td>
<td>32</td>
</tr>
<tr>
<td>Ni-21.5at%Cr</td>
<td>16</td>
<td>32</td>
</tr>
<tr>
<td>Cu-1.03at%Mn</td>
<td>41</td>
<td>16</td>
</tr>
<tr>
<td>Cu-3.3at%Mn</td>
<td>43</td>
<td>16</td>
</tr>
<tr>
<td>Cu-11at%Mn</td>
<td>30</td>
<td>16</td>
</tr>
<tr>
<td>Ti-5.2at%Al</td>
<td>Work-softening</td>
<td>5</td>
</tr>
</tbody>
</table>

Table 6.8: Work-hardening rates during single glide of Ni-Cr and Cu-Mn alloys as a function of solute content. (see text for details).

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Work-hardening exponent (n)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-5at%Al</td>
<td>0.33</td>
<td>34</td>
</tr>
<tr>
<td>Cu-10at%Al</td>
<td>0.22</td>
<td>34</td>
</tr>
<tr>
<td>Cu-15at%Al</td>
<td>0.12</td>
<td>34</td>
</tr>
</tbody>
</table>

Table 6.9: Work-hardening exponent as function of solute content in Cu-Al alloys. The data of Kugler et al[34] was fit to a Hollomon form between strain levels of 0.003 to 0.03 to get the n values. One can observe the n values to decrease with increase in solute content due to promotion of SRO. But comparing these n values with those presented for titanium alloys in table 5.1, the titanium alloys exhibit abnormally low n values.
<table>
<thead>
<tr>
<th>Heat treatment</th>
<th>Friction stress ($\tau_F$) (MPa)</th>
<th>$\gamma_2^{SRO}$ (mJ/m²)</th>
<th>$\gamma_1^{SRO}$ (mJ/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IWQ-350C-100h</td>
<td>133</td>
<td>30</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td>19</td>
<td></td>
</tr>
<tr>
<td>AC-1</td>
<td>186</td>
<td>45</td>
<td>$\approx$45</td>
</tr>
<tr>
<td></td>
<td></td>
<td>34</td>
<td>$\approx$34</td>
</tr>
<tr>
<td></td>
<td>166</td>
<td>40</td>
<td>$\approx$40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>$\approx$28</td>
</tr>
<tr>
<td>SQ600C-24h-400C-5h</td>
<td>155</td>
<td>36</td>
<td>42</td>
</tr>
<tr>
<td></td>
<td></td>
<td>25</td>
<td>31</td>
</tr>
<tr>
<td></td>
<td>167</td>
<td>40</td>
<td>46</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>34</td>
</tr>
<tr>
<td></td>
<td>195</td>
<td>48</td>
<td>54</td>
</tr>
<tr>
<td></td>
<td></td>
<td>37</td>
<td>43</td>
</tr>
</tbody>
</table>

Table 6.10: Diffuse APB energy on the prism plane was estimated in an IWQ-350C-100h, AC and a SQ-600C-24h-400C-5h sample using the "hair-pin" structures. Two different values of $\tau_{ss}$ was used based on single crystal work (69MPa) and polycrystal data (31MPa)( see text for details). From the observed microstructure, $\gamma_1^{SRO}-\gamma_2^{SRO}$ was estimated to be 6mJ/m² for the SQ-600C-24h-400C-5h sample and $\gamma_2^{SRO} \approx \gamma_1^{SRO}$ in the case of the AC sample.
Table 6.11: $\gamma_{SRO}$ on a prism plane estimated from pile-up tip stresses in a SQ-600C-24h-400C-5h sample. S, denotes a single ended pile-up and D, denotes a double ended pile-up. The estimated $\gamma_{SRO}$ values are in agreement with those estimated using the “hair-pin” structures and are comparable to those estimated in FCC-SRO alloys.
CHAPTER 7

OBSERVATION AND ANALYSIS OF WEAK-FRINGING (WF) FAULTS

7.1 Observation of weak-fringing faults

It was observed that weak fringes were present in slip bands between the dislocations in the band, indicating the presence of a fault. The weak-fringing faults (WF) were present in samples deformed under both constant strain rate and creep conditions. All the analysis presented here is on samples deformed in creep. Figure 7.1 shows the presence of these fringes in two samples with different heat treatments. In 7.1 (a) and (b) the presence of fringes in slip bands on basal and prism planes in a IWQ-350C-100h sample is shown. Fringes in slip bands on pyramidal planes are shown in figure 7.1(c); this was observed in a SQ-600C-24h-400C-5h sample. The fault fringes on the basal plane appear to be weaker than on both the prism and pyramidal planes. This was found to be true under all heat treatment conditions. Fringes were present in the slip bands of samples after all heat treatment conditions but was found to be weaker in the IWQ condition.

7.2 Nature of fault fringes

Figure 7.2 shows the g.R analysis of a fault in an AC sample. The dislocations present in the slip band bounding the fault are invisible for g = (0002) and g = (I101) and visible for 230
\( g = (10\overline{1}1) \) and \( g = (0\overline{1}11) \), indicating that the burgers vector of the dislocations is \( +/-a/3[11\overline{2}0] \). Slip trace analysis of the slip band indicated the slip plane to be \( (\overline{1}100) \). The fault fringes are also invisible when the dislocations are invisible. This indicates that the principal displacement (\( R \)) causing the fringing is "shear" type and \( R \) is \( +/-1/n[11\overline{2}0] \). The same WF fault (in a different region of the sample) was imaged under four different diffraction conditions namely, \( +g \)-bright field (BF), \( +g \)-dark field (DF), \( -g \)-BF and \( -g \)-DF and is shown in figure 7.3. The outer most fringes were observed to be symmetric in bright field (either bright/bright or dark/dark) and asymmetric in dark field. This contrast is similar to a stacking-fault contrast. According to theory, the outer most fringe is bright in BF if \( g.R \) is positive and vice-versa. Imaging under \( g = (10\overline{1}1) \)-BF, the outer most fringes are bright (figure 7.3 (a)), indicating \( g.R \) is positive. Hence, \( R = 1/n[11\overline{2}0] \) in the present case.

Figure 7.4 (a) shows the tip of a slip band in a SQ-600C-24h-400C-5h sample. The fault fringe contrast is absent or very weak between the first few dislocations and the contrast is clearly visible beyond 15-20 dislocations as one moves along the band. This indicates that the magnitude of the fault vector \( R \) is increasing along the band. Suggesting that the fault vector \( R \) increases in magnitude with each shearing event experienced by a particular area of the slip plane. In 7.4 (b) a region near a dislocation source (which has been "cut off" due to the foil preparation) is shown. The region near the source is indicated as \( S \) in the figure. It appears that this particular source emitted only five dislocation loops, as evidenced by the five pairs of screw dipoles present. (Note: Compare the lobe contrast of the dislocations to one side of the source versus the other side. One can observe that they are opposite in character). The fault fringes, between the fifth pair, are barely visible above the background. This region should have experienced a
shear equivalent to five times the magnitude of the burgers vector. This again indicates that the fault vector magnitude depends on the number of dislocations that have sheared a particular region of the slip plane and is very small in this case. Another general observation regarding the fault fringes is that the fault contrast appears very uniform further along the band after many dislocations.

In some cases, it was observed that there was residual fringe contrast when the dislocations contained in the fault plane were invisible. This implies that the displacement vector \( \mathbf{R} \) has an out of plane component in addition to the principal in-plane shear component. Figure 7.5 (a) shows such an example from an IWQ-650C-24h sample. The dislocations in the slip band are invisible but there is a strong residual fringe contrast from the fault. Burgers vector of the dislocations in the slip band was determined to be \( a/3[\overline{1}2\overline{1}0] \) from \( \mathbf{g}, \mathbf{b} \) analysis. Trace analysis of the slip band indicated that some cross-slip had occurred in this band and the overall slip plane was an irrational plane. The slip plane was close to \((10\overline{1}0)\) prism plane and the basal plane could be the cross-slip plane. It was also observed that fringes were completely invisible when imaged with \( \mathbf{g}=(0002) \). This indicated that \( \mathbf{R} \) is \((+/-1/n_1[10\overline{1}0])+(+/-1/n_2[\overline{1}2\overline{1}0])\). From this one might interpret that whenever there is cross-slip taking place in the slip band, the fault fringes had an out of plane component in addition to the in plane shear component. But, this is not the case. Figures 7.1 (c) and 7.5 (d) show two slip bands labeled A and B in a SQ-600C-24h-400C-5h sample. Slip band A is a prism slip band, \( a/3 [\overline{2}110](01\overline{1}0) \), and band B is a pyramidal band, \( a/3 [\overline{2}1\overline{1}0](01\overline{1}1) \). For \( \mathbf{g}=(0\overline{1}11) \), the dislocations in both bands are invisible. The fault fringes are clearly invisible in band A, but there is some weak residual contrast in band B (indicated by the arrow in figure 7.5 (d)). Clearly there is some cross-slip at the tip of band A, but there is no residual fringe contrast. It should also be noted that the
residual fringe contrast in the SQ-600C-24h-400C-5h sample is much weaker than the contrast from the fault in the IWQ-650C-24h sample (figure 7.5a). The other possibility is that samples aged above the \( \alpha/\alpha_2 + \alpha \) transus (which is approximately 550°C for a Ti-6wt% Al composition [1, 2]) and subsequently deformed always have faults with an out-of-plane component. Figures 7.5 (b) and 7.5 (c) show two slip bands, labeled C and D in a SQ-600-24h sample. Slip band C is a prism band, \( \alpha/3 [\bar{2}110](01\bar{1}0) \) and D is another prism band, \( \alpha/3[\bar{T}2\bar{T}0](10\bar{T}0) \). It is clear from the two images that band C shows no residual fringe contrast, whereas band D does show significant residual fringe contrast. Finally, it was observed in all the cases that there was no fringe contrast when imaged with \( g=(0002) \).

From the above discussion, it appears that there is some normal component to the fault vector \( \mathbf{R} \), and the magnitude of the normal component is dependent on the amount of shear experienced in a particular region of the slip plane. It is possible that the normal component is always stronger in the case of samples aged above the \( \alpha/\alpha_2 + \alpha \) transus, but evidence for that is not conclusive. To reiterate, the normal component is negligible in the AC sample and in the samples aged below the \( \alpha/\alpha_2 + \alpha \) transus.

### 7.3 Analysis of fault character and magnitude using Cufour

There were many observations that were made regarding the nature of the weak-fringing faults in the last section. We used the image matching technique to understand some of the characteristics of the fault fringes. Images were computed for comparison with the experimental images using the program Cufour developed by Schäublin and Stadelmann [3]. Cufour version 2.1, which is capable of handling hexagonal crystal system, was run on an Apple G3 Power Macintosh computer to calculate the images. The concept of a generalized cross-section developed by Head et al[4] is used in Cufour. This restricts the
program to simulate dislocation lines that are parallel and inclined in the thin foil. Since in all of our observations, the preferred line orientation for the dislocations was observed to be along the screw direction, this restriction is not a serious limitation. Cufour uses anisotropic elasticity to calculate the defect displacement field using the Ancalc subroutine of Head et al.[4]. For our calculations, single crystal elastic constants of pure titanium at room temperature was used. These data were taken from the work of Fisher and Renken[5] and are listed in table 7.1. In the Cufour program the defect displacement field inside an infinite crystal is used to describe the defects in a thin foil, hence, surface effects are not included. In our calculations, images were calculated with foil thickness greater than $5\xi_a$ (main beam), hence, surface effects should be minimized. The projected line direction of the dislocations are from left to right of the image in the simulations and the top of the foil is always on the right side of the image. In some cases the simulated image had to be rotated to be in correspondence with the experimental image. The projected line directions are shown in all the calculated images to avoid any confusion.

For all calculations five beams namely, -2g, -g, 0, +g, +2g were used for computing the image. These and other input parameters common for all the calculations are listed in table 7.1. The lattice parameter for Ti-6Al was taken from the work of Clark et al.[6]. It is important to note that in all simulations only the shear component of the fault vector was used. The out of plane component of the fault vector was ignored. This is reasonable, because in majority of the cases the out of plane component was very small or not present.

It was observed in the experiments that the fault contrast on the basal plane was always weaker than the contrast on both prism and pyramidal planes. Figure 7.6 shows two images computed using Cufour with the same fault on a basal (00001) and on a prism
(T100) plane. The geometry and other input parameters used for the calculation are listed in table 7.2. Contrast of the fringes appear very similar in both images, and are quantitatively of the same magnitude in terms of dark bright contrast. Therefore, the magnitude of the fault vector on the basal plane truly seems to be much smaller than the magnitude in both prism and pyramidal planes.

Quantitative image matching was performed to estimate the magnitude of the fault vector causing the fringing in an AC sample. To be able to do quantitative matching of intensities between the experiment and simulations one needs to know the experimental intensities accurately. In order to avoid the non-linearity of photographic emulsions, images of the faults were acquired using a CCD camera in Gatan imaging filter (GIF) system attached to a Philips CM200 TEM. Zero loss electrons of 197 keV, within a window of 20eV were used to form the images. The acquired images were corrected for both the flat-field and the dark-field response of the CCD array. Images under four diffraction conditions namely, +g-BF, +g-DF, -g-BF and -g-DF were obtained using the CCD camera. Two images were acquired under each condition, one image with the fault covering the majority of the field of view and another image with the background covering significant portion of the field of view. Figure 7.7 shows a typical CCD image of the fault (taken under -g-DF imaging conditions). In the experimental images, the foil thickness is changing continuously. This causes the fault width to change continuously. Further, one needs to average intensities over some area to get reliable intensity values. For consistency, intensities were measured from the same area in all the images. (Note: All intensity measurements, both from experiments and simulations were made on the bottom side of the foil). The average intensities measured from the two CCD images (for each diffraction condition) were taken as the experimental intensities. No reference
images were taken to measure the incident electron dose. But, in the simulations, the image intensities are normalized to the incident electron intensities. Hence, only ratios of intensities were compared between simulation and experiment.

In the simulations, images with one dislocation along the screw orientation with a fault on the slip plane were calculated. The procedure adopted for quantitative image matching is as follows, the foil normal, dislocation burgers vector, slip/fault plane and the fault vector direction were determined experimentally. These parameters are listed in table 7.3. The projected width of the fault was measured in the experimental images (≈675nm) and the number of dark fringes in the fault were counted. Using these two criteria, the foil thickness for the simulation was fixed at 535nm, such that the number of dark fringes and the projected fault width matched the experimental images. To estimate the experimental diffraction conditions as accurately as possible, the ratio of the maximum intensity of bright lobe of the dislocation to the background intensity was compared between experiment and simulation. For the dislocation bright lobe intensity measurements, an average over a 10nm$^2$ area around the brightest pixel of the bright lobe was taken from the experimental images. Similarly, an average over a 10nm$^2$ area around the brightest pixel of the bright lobe was taken from the simulated images. Care was taken to average intensities only on the bright side of the lobe. The experimental background intensities were obtained by averaging over an area of between 55nmx100nm to 330nmx100nm at a distance of 150nm from the dislocation and 30nm from the fault. Background intensities were obtained from the simulated images by averaging over an area of 285nmx100nm at a distance of 150nm from the dislocation and 30nm from the fault. The anomalous absorption parameters and the deviation parameter (w) were changed in the simulations until the optimum fit of the ratio of the maximum intensity of bright lobe of the
dislocation to the background intensity \( \frac{I_{\text{max}}}{I_{\text{background}}} \) between experiment and simulation for all four diffraction conditions was obtained. With this procedure all the parameters needed for the simulation were fixed. One can now change the magnitude of the fault vector \( \mathbf{R} \) and match it with the experimentally observed contrast.

For the fault fringe intensity comparison, measurements were made only on the first dark and bright fringe on the bottom side of the foil. Again, for consistency, measurements were made at the same location in all the images. The fringe intensities were measured at a distance of 150nm from the dislocation and averaged over a height of 100nm (shown by the rectangle in figure 7.7). Fringe intensities were measured by the same procedure in the calculated images. A parametric analysis was done to determine the most sensitive parameter that can be used to match the calculated and experimental image fringe intensity ratios. Figure 7.8 shows a plot of \( n \) versus intensity ratios from images calculated for various magnitudes of fault vector \( \mathbf{R} \). \( 'n' \) represents the magnitude of \( \mathbf{R}(1/n[11\bar{2}0]) \); larger \( n \)-values correspond to smaller \( \mathbf{R} \)-values. Three intensity ratios are plotted in the figure as a function of \( n \). They are: (a) ratio of the maximum of the bright fringe intensity to minimum intensity of the dark fringe \( \frac{I_{\text{fringe max}}}{I_{\text{fringe min}}} \), (b) maximum of the bright fringe intensity to the background intensity \( \frac{I_{\text{fringe max}}}{I_{\text{background}}} \) and (c) background intensity to the minimum intensity of the dark fringe \( \frac{I_{\text{background}}}{I_{\text{fringe min}}} \). This plot shows that \( \frac{I_{\text{fringe max}}}{I_{\text{fringe min}}} \) is the most sensitive parameter to the magnitude of \( \mathbf{R} \), particularly at smaller values of \( n \). Image matching was done by choosing the \( \mathbf{R} \) value that best fit the
experimentally observed ratio $\frac{I_{\text{fringe}}}{I_{\text{max}}}$ (log-log scale). The best fit simulation parameters along with the best match $R$ values are listed in Table 7.4. Figure 7.9 shows the simulated images for the best fit $R$ values listed in Table 7.4. These images can be qualitatively compared with the experimental images shown in Figure 7.3 (which were captured in photographic plates). Note that the simulated and experimental images cannot be compared quantitatively, because the contrast of the experimental images have been altered for presentation.

The estimated $R$ values for the different diffraction conditions seem to have significant spread in magnitude. The best fit of experimental conditions from the intensity matching of the bright lobe of the dislocation seem to be for the $+g$-DF condition and the $R$ value estimated for this condition is probably the most reliable. There are a few possible reasons for the apparent poorer matching of the intensity ratios of the dislocation lobe between experiment and calculations. One reason could be effect of foil bending, not included in the simulations. The effect of foil bending can be seen in the variation of background intensity in the experimental images (Figure 7.3). This affects the experimental intensity ratio, hence, affects the fit. The second reason could be the use of elastic constants of pure titanium in the calculation of defect displacement field. One would expect this to affect the details of the dislocation lobe contrast, but the calculated images do correspond well with the observed experimental dislocation lobe contrast. This suggests that the use of elastic constants of pure titanium is a reasonable approximation. Finally, Figure 7.8 clearly shows that it is difficult to estimate the magnitude of $R$ with confidence beyond an $n$-value of approximately 100. The fact that the contrast is similar for $R$ values beyond $n=100$ could also explain the apparent uniform contrast of the fault fringes observed over large distances in the middle of a slip band. It should be pointed out that as the magnitude of $R$ decreases, the fringes in the middle of the foil will be
damped severely and will be missing for very low values of \( R \). In the present images the fringes in the middle of the foil are clearly visible. Taking into account all the above factors, the estimated values of \( R \) are within reasonable limits and probably close to the actual magnitude of \( R \).

It was shown in the previous section that the residual displacement causing the fringes have a displacement primarily in the slip plane. One important question that needs to be answered is whether the direction of residual displacement is along the direction of slip or is opposite to the direction of slip. In other words, it is important to know whether the magnitude of the defective dislocations \( |b+R| \) are larger or smaller in magnitude compared to a lattice translation along \(<11\bar{2}0>\). To determine this information one needs to know the actual direction of slip (i.e. the sense of shear) in the slip plane. An example of the determination of the sense of shear in the slip plane is shown in figure 7.10 for an AC sample. Figure 7.10 (a) shows two expanding dislocation loops. For convenience, only the screw and edge components are shown. The two loops have opposite sense of shear with respect to each other. In case(1), the top dislocation is a positive screw dislocation and in case(2), the bottom dislocation is a positive screw dislocation. These are the two possible configurations that one would encounter and it shows that if one can identify the orientation of the screw dipole (which is of interest in our case), we can in fact determine the sense of shear in the slip plane. Figure 7.10 (b) shows the location of such a screw dipole in an AC sample. In fact, the extrapolated position of the source as indicated is based on the fact that all the dislocations to either side of this position are opposite in character (this is not shown in the picture, but it has been observed that the dislocations above dislocation \( d_i \) have the same sign as \( d_i \) from the dislocation lobe contrast). Using Cufour, choosing a particular line direction for both dislocations,
\( u = [1\overline{1}20] \), the lobe contrast of the screw dipole was matched to determine the orientation of the positive and negative screw dislocations forming the dipole. The two calculated images are shown in figures 7.10 (c) and 7.10 (d). The input parameters used for calculating the images are listed in table 7.5. Comparing the lobe contrast of the dislocations in the two calculated images with the experimental image, one can see that image 7.10 (c) matches the experimental image. It is also clear that the experimental image corresponds to case (1) shown in figure 7.10 (a). This indicates in the present case that the sense of shear is along \([1\overline{1}20]\). In other words the sense of shear in the slip plane \((+b)\) and the residual displacement \((+R)\) causing the fringing are parallel to each other. Therefore, the magnitude of the dislocations are effectively larger than a lattice translation along \(<1\overline{1}20>\). Similar analysis was performed in a SQ-600C-24h-400C-5h sample and it was found that the sense of shear of the dislocations and the residual displacement vector were also parallel, and appears to be true in all the cases that have been observed in this work.

### 7.4 In-situ heating experiments

In-situ heating experiments were conducted in a CM 200 TEM using Gatan model 652 hot stage holder. Figure 7.11 shows images of a fault in a IWQ-350C-100h sample during in-situ heating and after subsequent cooling to room temperature. Two parallel slip bands on a prism plane \((\overline{1}100)\) are shown (A and B). The same region has been imaged before in-situ heating and slip band A is shown in figure 7.1 (b). Two dislocations, \(d_1\) and \(d_2\) in slip band A, have been identified in all the images as a point of reference. The samples were ramped up to different temperatures and allowed to equilibrate at that temperature for few minutes and images of the fault were taken. Figure 7.11 (a) shows the fault imaged at 350°C. Comparing this image with figure 7.1 (b), it appears that the fault
contrast has not diminished significantly between room temperature and 350°C, taking into account the fact that image 7.1 (b) was imaged at room temperature. (Note: The grainy appearance of the background is due to the oxidation of the foil during in-situ heating inside the microscope). The fault was imaged at 412°C after the sample was annealed at this temperature for approximately 10 minutes. This is shown in figure 7.11 (b). Here the fault contrast has almost disappeared, indicating that the fault has been annealed-out. The sample was then cooled to room temperature. It was removed from the microscope and was cleaned in a low energy plasma cleaner to remove some of the oxide layer. The same region was imaged again at room temperature after the cleaning process. Figure 7.11 (c) shows the fault imaged at room temperature after the in-situ anneal treatment. The fault contrast is definitely very weak after the anneal treatment, although there is some faint contrast. This was true for all the faults present in the sample. This demonstrates that the fault can be annealed out by subsequent heat treatment, although in the present case it appears that the annealing time was not sufficient to completely anneal out the fault. It is worth reiterating that there was no significant decrease in the fault contrast until about a temperature of 350°C. The above results demonstrate that indeed the fault can be annealed out at temperatures where atomic rearrangement are possible.

7.5 Origin of residual displacement causing the weak fringes

In the literature this kind of weak fringe contrast has been observed in slip bands in some fcc alloys[7, 8]. Clarebrough [7]observed weak fault fringes between dislocation in a Cu-15at% Al alloy. He noted that the fault vector causing the fringing was normal to the slip plane, \( \mathbf{R} = \frac{1}{n} \langle 111 \rangle \). Using image matching simulations, he further noted that the magnitude of the fault vector was between \( \frac{1}{150} \langle 111 \rangle \) to \( \frac{1}{200} \langle 111 \rangle \). Destruction of short-range order (SRO) was proposed to cause unfavorable Al-Al nearest neighbors in
the slip plane, leading to a residual dilation of the slip plane causing the weak fringes in the slip plane. Further, Clarebrough performed ex-situ annealing experiments on TEM foils and noted that the fringes disappeared after an anneal of 1h at 300°C. Brooks et al [8] observed weak fault fringes in a Pd-9.4at% Ce alloy between dislocations on <111> slip planes. Their experiments showed that the fringing was due to a residual displacement in the slip plane indicating that \( R = 1/n <110> \). Again, the cause for this residual displacement was attributed to destruction of SRO of Pd and Ce atoms. It was proposed that due to the large size difference of Pd and Ce atoms, the defective dislocations caused a displacement less than a lattice translation. It should be noted that no evidence was provided in the work to show that the dislocations in fact produced a displacement shorter than a lattice translation along <110>. Similar weak-fringing faults were observed by Clarebrough in a Al-10at% Ni alloy[9]. He noted that the faults had a displacement \( R = 1/n [110] \) and that the dislocations produced a displacement larger than the lattice translation along [110]. This result is similar to our observations.

These weak-fringing faults have been observed in many works on titanium alloys. It has been observed in single phase alloys[10, 11] and in the alpha phase of two phase alloys as well[12-15]. Weak fringes were observed in both prism and pyramidal planes. In all of these works, the fringes are present in published micrographs but no reference is made to the fault fringes except for that of Woodfield[15]. Recently, similar faults have been observed in a \( \gamma \)-TiAl alloy[16]. In that work it was attributed to the presence of SRO of a Ti\(_3\)Al phase. Woodfield analyzed these weak-fringing faults in a Ti-5331S alloy and observed that the displacement causing the fringing was normal to the prism slip plane, \( R=1/n<01\overline{1}0> \). He did not observe the faults on the basal plane. He also noted that the fault fringes were not present in all the prism slip bands. It was argued that the residual
displacement is produced due to destruction of SRO of Ti-Al atoms. This was thought to cause unfavorable nearest neighbor configurations and induce residual displacement normal to the slip plane. In order to explain the absence of fault fringes in the basal plane, he proposed a model in which it was argued relaxation in the elastically stiffer basal planes is more difficult than in the elastically softer prism planes. Further, to explain the apparent absence of fringes on all prism planes, he argued that in the ordered Ti₃Al (DO₃) structure, there are two kinds of prism planes. In type I plane, no nearest neighbor violations occur and in type II 2 2/3 nearest neighbor violations occur due to the lattice translation. In the ordered structure it is expected that the dislocations will move in type I planes, because moving on type II planes will cause an anti-phase boundary. In a small SRO region, the difference in energy between passing a dislocation on a type I plane versus a type II planes will be considerably less than in Ti₃Al. It was argued further that if the energy for cross-slip is greater than the energy of the anti-phase boundary caused on a type II plane when cutting an SRO region, then slip will continue on the type II plane. Hence, whenever the slip plane intersects SRO volumes, the slip plane will be subject to some displacement when it cuts such regions. This will cause an overall displacement perpendicular to the whole slip plane. Also when the slip plane encounters part of the matrix with no SRO or little SRO no fringing will be expected on those planes.

The observation of Woodfield[15], that there are no faults observed on basal planes is in apparent conflict with our results, where we have clearly observed these faults on basal planes (figure 7.1(a)). It should be reiterated again that in our case the fault fringes on the basal planes are always weaker than the faults on prism and pyramidal planes. Further, we notice that the fault fringes are best imaged under an s=0 (i.e. on bragg diffraction condition) in both bright field and dark field. The fault fringes are very weak on basal
planes and do not image clearly if one deviates from the $s=0$ condition. This could possibly explain the absence of basal fault fringes in the work of Woodfield. The second result that seems to be in conflict with our observations is that he does not observe fault fringes on all prism slip bands. In our observations the fringe contrast is present in all $<a>$ type slip bands on all slip planes. In the work of Pelissie et al. [14] on Ti-6Al-4V, the fault fringes are present in all the parallel prism slip bands in the alpha phase (see their figure 4a). Further, Woodfield’s model would suggest that on the same slip plane one could have fringes in some regions and no fringes in other regions of the plane. We have explained in detail in section 7.2 that the residual displacement causing the fringing seems to be additive with the number dislocations shearing a particular region of the sample. Hence, the fringe contrast is very weak in regions where only a few dislocations have sheared the slip plane. Again, if one were to image these slip bands in a bright-field diffraction condition with a positive deviation from bragg condition, the fringes will not be imaged very well and could be interpreted as being absent. This situation can be seen figure 7.2 (a). From looking at that image one can easily conclude that there are no fringes present in bands B and D and in fact the fringes in band A appear very weak, while we know from other observations that fringes are present in all the slip bands. The principal difference between Woodfield’s[15] observations and our results is in the nature of the residual displacement itself. We observed that the primary residual displacement to be in the slip plane and along the burgers vector. Woodfield observed residual displacement normal to the slip plane. In our observations the normal component is stronger in samples aged above the $\alpha/\alpha_t+\alpha$ transus and is very weak or absent in samples aged below the transus. The normal component of the displacement vector was found to be the strongest in the IWQ-650C-24h sample. Our results would suggest that if one were to age the samples above the $\alpha/\alpha_t+\alpha$ transus one might induce an SRO state which
strengthen the residual displacements normal to the slip plane to such an extent that they exceed the magnitude of displacement in the slip plane. We do not know the heat treatment history of Woodfield's sample. It is possible that Woodfield's sample underwent such a treatment above the transus, hence, had a residual displacement primarily normal to the slip plane. The other possibility is that the nature of SRO was different in Woodfield's sample because of other alloying elements.

In chapter 4, we have presented neutron diffraction evidence for the presence of SRO of Ti-Al atoms after all the heat treatments in our material. The in-situ heating experiment results presented in section 7.4 showed that the faults can be annealed out at temperatures when atomic rearrangements are possible. Considering the above factors, it is reasonable to suggest that the residual displacement causing the fringing is due to destruction of SRO leading to unfavorable Ti-Ti or Al-Al nearest neighbors. It is indeed possible that the SRO state is different when the sample is aged above the $\alpha/\alpha_2+\alpha$ transus, compared to samples aged below the transus or in an AC condition. The 'raw' neutron diffraction data presented in chapter 4 does not seem to support this hypothesis. But detailed fourier decomposition analysis have yet to be done on this data to determine the Warren-Cowley SRO parameters. It is possible that subtle differences in SRO state do exist which can be deduced only from the SRO parameters. The detailed fringe contrast analysis presented in the previous sections, would suggest that the nature of fault fringes might actually be a more sensitive indicator of SRO state than even neutron scattering experiments. At present there is no microstructural model available for SRO in hexagonal materials especially for the Ti-Al system. Without this information one cannot propose a detailed atomistic model for the origin of residual displacements.
7.6 Summary

(a) Weak-fringing faults were observed in <a> type slip bands between dislocations on basal, prism and pyramidal planes. These fringes were present in samples that were deformed in both constant strain rate conditions and in creep. All the heat treatments produced these fault fringes in deformed samples and it was the weakest in an IWQ sample.

(b) The residual displacement causing the fringing was determined to be primarily in the slip plane. The maximum magnitude of $\mathbf{R}$ was evaluated using quantitative image matching simulations for an AC sample. It was estimated to be in the range of $1/104[1120]$ to $1/145[1120]$. Further, using image matching simulations it was determined that $\mathbf{R}$ is parallel to the burgers vector, indicating that the dislocations cause an effective displacement larger the lattice translation along <1120>

(c) The normal component of the residual displacement vector $\mathbf{R}$ was found to be very small or absent for samples aged below the $\alpha/\alpha_2+\alpha$ transus as well as for the AC condition. In samples aged above the $\alpha/\alpha_2+\alpha$ transus the normal component was significant and was found to be the maximum for an IWQ-650C-24h sample.

(d) The displacement $\mathbf{R}$ was found to be additive with the number of dislocations shearing a particular region of the slip plane. The fault fringes were found to be absent or weak near the tip of a slip band and the contrast of the fringes increase as one moves along the slip band.

(e) Fault fringes on basal planes were observed to be very weak. Image simulations using Cuffour indicated that the weak contrast is not a dynamical contrast effect. It appears that magnitude of the fault displacement vector is always low on the basal planes.
(f) In-situ heating experiments indicated that the residual displacement fault can be annealed out, by annealing the sample at temperatures where atomic rearrangements are possible.

(g) Considering the presence of SRO in this alloy, it is suggested that destruction of SRO leads to unfavorable Al-Al or Ti-Ti nearest neighbors. This causes the residual displacement leading to fringing in the slip bands.

(f) Detailed analysis of the faults suggest that the fringe contrast might be a more sensitive indicator of the SRO state in the sample compared to neutron scattering experiments.
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Figure 7.1: (a) Shows the presence of weak fringes between the dislocations in a slip band on basal plane in a IWQ-350-100h sample. The sample was deformed in creep. (b) In the same sample, fringes are present in a slip band on prism plane. (c) Presence of weak fringes between the dislocations in a slip band on pyramidal plane is shown here. This was observed in a SQ-600-24h-400-5h sample deformed in creep.
Figure 7.2: $g \cdot R$ analysis of the fault fringes is shown here. The fault and the dislocations are visible in (a) and (b). Four parallel slip bands with $<a>$ type dislocations have been identified with labels A through D. Band E is a band of $<c+a>$ dislocations. These dislocations can be used to identify the location of the different $<a>$ slip bands. The dislocations and the fault fringes are invisible in (c) and (d). This shows that the fault vector causing the fringes is primarily 'shear' in character. The burgers vector $b$ of the dislocations and the fault vector $R$ is $\pm 1/3 [11\overline{2}0]$. Additional slip trace analysis indicated that the slip plane is $(1\overline{1}00)$. In (b) $g \cdot R$ is negative for the fault (this can be clearly seen in band A), because the outermost fringes are dark in bright field. Hence, the $R$ value is $1/n [11\overline{2}0]$ for the fault.
Figure 7.3: WF fault in an AC sample (same as in figure 7.2) was imaged under four diffraction conditions +/- g BF/DF, shown in a-d. The outer fault fringes were symmetric in bright field (either dark/dark or bright/bright), but asymmetric in dark field. This fringe contrast is similar to stacking-fault contrast. The residual displacement causing the contrast is $R = 1/n [1120]$. 

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Figure 7.4: Tip of a slip band in a prism plane is shown in (a). The fault contrast is very weak at the tip. The contrast get stronger along the band and is easily visible after 15 dislocations from the tip. A source, the indicated position of which (S) has been extrapolated parallel to the screw orientation, which has emitted 5 loops (only the screw segments are present) is shown in (b). The fault contrast is just visible in between the fifth pair (noted as WF). It clearly demonstrates that the fault vector causing the fringing is increasing in magnitude with the number dislocations shearing a particular region. Both the observations were made in a SQ-600-24h-400-5h sample after deformation in creep.
Figure 7.5: In (a) fault fringes in a IWQ-650-24h sample is shown. The dislocations present in the slip band are invisible. There is a strong residual fringe contrast from the fault indicating that there is significant out of plane component to the fault vector $\mathbf{R}$. Slip bands in a SQ-600-24h sample have been imaged under two diffraction conditions in (b) and (c). The dislocations in band C are not visible and there is no residual fringe contrast from the fault in (b). Also slip band D and the fault on band D is visible. In (c) band C is visible, but band D is invisible and there is a residual contrast from the fault fringes. Slip bands in a SQ-600-24h-400-5h sample is shown in (d). This is the same region shown in 7.1(c), under a different diffraction condition. Here slip bands A and B are not visible, there is residual fringe contrast in band B and not in band A.
Figure 7.6: Faults on basal (a) and prism planes (b) were simulated using Cufour under the same diffraction conditions and magnitude of the fault vector $\mathbf{R}$. Simulations parameters are listed in table 7.2. Comparison of the contrast between (a) and (b) shows that the fringe contrast is similar on both planes. The contrast comparison indicates that the weak contrast observed on basal planes in the experiments is due to the real nature of the fault magnitude.
Figure 7.7: Shown above is an example of a fault image in an AC sample that was captured using a CCD camera. The top and bottom of the foil are indicated as T and B respectively. The image intensity of the first dark and bright fringes were measured about 150 nm from the dislocation and was averaged over a height of 100nm (the region inside the box).

Figure 7.8: A plot of intensity ratios versus n obtained from Cufour simulations. The plot shows that ratio of maximum of the bright fringe to the minimum of the dark fringe is the most sensitive parameter for comparison between simulation and experiment, particularly at low values of n.
Figure 7.9: a-d shows the images calculated using Cufour that were quantitatively matched with the experimental images shown in figure 7.3. (Note: The contrast of the images in figure 7.3 has been modified and cannot be compared with the simulated images quantitatively). Simulation parameters for the four conditions are listed in Table 7.3. The projected line direction is shown as \( u_p \) and the true line direction for the dislocation is [1120]. The top and bottom of the foil is indicated in (b). See text for details. (continued)
Figure 7.9: continued.
Figure 7.10: (a) Two expanding loops with only edge and screw components are shown. The burgers vector $\mathbf{b}$ ('shear') for the two cases are opposite. In case (1), the top dislocation segment has a positive screw orientation, and in case (2), the bottom dislocation segment has a positive screw orientation. (b) Shown is the relative location of the dislocation source (S) in an AC sample. The slip plane is $\{1\overline{1}00\}$, the burgers vector of the dislocation dipole is $\pm 1/3[11\overline{2}0]$ and the fault vector causing the fringes is $1/n[11\overline{2}0]$ for this slip system. The two dislocations forming the dipole ($d_1$ and $d_2$) are near screw orientation. (c) and (d) are images calculated using Cufour. (The simulation parameters are listed in table 7.5). The line direction was chosen as $[11\overline{2}0]$ for the two dislocations and the burgers vector for the two dislocations were chosen to match the lobe contrast of the observed dislocation pair. It is clear that image (c) matches the experimental image, indicating that slip is along $[11\overline{2}0]$ and belongs to case (1). This shows that the residual displacement $R$ causing the fault fringes is along the burgers vector.
Figure 7.11: Shown above are images of a fault in a IWQ-350-100h sample during and after in-situ heating. The fault is the same as the one shown in figure 7.1(b). Two slip bands have been identified as A and B. Also, two dislocations $d_1$ and $d_2$ in band A have been labelled for reference in all three images. The fault is clearly visible at 350°C, but is very weak at 412°C. The sample was held at 412°C for approximately 10 min and then cooled room temperature. The fault is very weak at room temperature after the in-situ anneal treatment as shown in (c). This indicates that the fault formed due to slip can be annealed out by subsequent heat treatment. Note: The grainy appearance of the background is due to the oxidation of the sample during the in-situ heating experiment in the microscope.
Material: Ti-6wt% Al; Crystal Structure: hcp; Space group: P6/m/mc(#194)
a=0.2931 nm, c=0.4676 nm [6]
Elastic Constants: c_{11}=162.4, c_{12}=92.0, c_{13}=69.0, c_{33}=180.7, c_{44}=46.7, c_{66}=35.2 GPa [5]
Electron Beams: -2g, -g, 0, +g, +2g
Extinction distances: \( \xi_{(10\bar{1}1)} = 60.79 \), \( \xi_{(2\bar{2}1)} = 253.7 \) nm for 200 kV electrons
Extinction distances: \( \xi_{(10\bar{1}1)} = 60.52 \), \( \xi_{(2\bar{2}1)} = 252.6 \) nm for 197 kV electrons

Table 7.1: Common input parameters used in Cufour simulation.

<table>
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<th>Parameter</th>
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</thead>
<tbody>
<tr>
<td>Acceleration Voltage</td>
<td>200 kV</td>
</tr>
<tr>
<td>Anomalous absorption coeff.</td>
<td>( A_{110\bar{1}1} = 0.025 ), ( A_{120\bar{2}2} = 0.044 ) (calculated using Cufour)</td>
</tr>
<tr>
<td>Foil normal</td>
<td>[3962]</td>
</tr>
<tr>
<td>Foil thickness</td>
<td>400 nm</td>
</tr>
<tr>
<td>Fault plane normal</td>
<td>[0001] and [\bar{1}100]</td>
</tr>
<tr>
<td>Fault vector direction and magnitude</td>
<td>( R = 1/9[11\bar{2}0] )</td>
</tr>
<tr>
<td>Diffraction Conditions</td>
<td>( g = (01\bar{1}1); \ B = [21\bar{3}] ); ( w = 0 )</td>
</tr>
</tbody>
</table>

Table 7.2: Simulation input parameters for contrast comparison between basal and prism faults.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acceleration Voltage</td>
<td>197 kV</td>
</tr>
<tr>
<td>Anomalous absorption coeff.</td>
<td>( A_{110\bar{1}1} = 0.06 ), ( A_{120\bar{2}2} = 0.06 )</td>
</tr>
<tr>
<td>Foil Normal</td>
<td>[2750]</td>
</tr>
<tr>
<td>Foil thickness</td>
<td>535 nm</td>
</tr>
<tr>
<td>Fault plane normal</td>
<td>[\bar{1}100]</td>
</tr>
<tr>
<td>Fault vector direction and magnitude</td>
<td>( R = 1/n[11\bar{2}0] )</td>
</tr>
<tr>
<td>Diffraction Conditions</td>
<td>( +g = (10\bar{1}1) ) and ( -g = (\bar{1}0\bar{1}1); \ B = [21\bar{3}] )</td>
</tr>
</tbody>
</table>

Table 7.3: Input parameters for quantitative image matching simulations.

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Table 7.4: Comparison of experimental and best match simulation intensity ratios.

<table>
<thead>
<tr>
<th>Diffraction Condition</th>
<th>$\frac{I_{\text{lobe}}}{I_{\text{background}}}$</th>
<th>$\frac{I_{\text{fringe max}}}{I_{\text{fringe min}}}$</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>$+gBF$; w= +0.078</td>
<td>1.34</td>
<td>1.25</td>
<td>132</td>
</tr>
<tr>
<td>$+gDF$; w= -0.078</td>
<td>1.54</td>
<td>1.24</td>
<td>118</td>
</tr>
<tr>
<td>$-gBF$; w= +0.078</td>
<td>1.14</td>
<td>1.23</td>
<td>145</td>
</tr>
<tr>
<td>$-gDF$; w= -0.078</td>
<td>1.44</td>
<td>1.305</td>
<td>104</td>
</tr>
</tbody>
</table>

Table 7.5: Input parameters for 'shear' determination simulations.

- Acceleration Voltage: 200 kV
- Anomalous absorption coeff: $A_{1:1011:}=0.025$, $A_{1:2022:}=0.044$ (calculated using Cufour)
- Foil normal: $[2750]$
- Foil thickness: 535 nm
- Fault plane normal: $[\overline{1}100]$
- Fault vector direction and magnitude: $R=1/99[11\overline{2}0]$
- Diffraction Conditions: $+g=(10\overline{1}1)$; $B=[\overline{1}2\overline{1}0]$; w=0
CHAPTER 8

ROOM TEMPERATURE DEFORMATION ANOMALIES IN TITANIUM ALLOYS

8.1 Negative creep in Ti alloys

8.1.1 Experimental results

It was shown in chapter 6 that titanium alloys deform in a very planar fashion in the α phase during plastic deformation both under creep and constant strain rate conditions. The deformation propagates through slip bands with hundreds of dislocations in them. These bands consist of large pile-ups of screw dislocations. We observed, that during creep if a complete load drop is made after some straining there was no backflow in the material. This is shown in figure 8.1. The sample was crept to a plastic strain of 1.6% at a stress level of 716 MPa and then completely unloaded. Absence of backflow upon complete unloading is very unusual because as mentioned above the deformation microstructure consists of numerous large pile-ups with many regions of high stress concentrations. Conventionally, one would expect some backflow with this microstructure after a complete load drop. What is even more interesting is that when we reload the material to the original stress level after such a load drop, the material creeps...
in a negative fashion for 10s of seconds before it creeps forward again. The magnitude of observed negative creep strains are of the order of 10-100 micro strains and this occurs for a duration of 10-100 seconds. We have observed this negative creep phenomenon in both the Ti-6Al and Ti-6242 alloys. An example of negative creep flow observed in Ti-6Al-DI is shown in figures 8.2. These are linear total strain versus time plots showing an unload/reload sequence. The magnitude of negative creep strains and the duration of negative creep response after various unload/reload experiments are listed in table 8.1. It also lists the forward creep rate immediately following the negative creep response. In one case, in a Ti-6242 alloy the sample was reloaded to a stress level of 90% of the creep stress. The data in table 8.1 seem to indicate that the duration of negative creep response increases as the forward creep rate decreases. It also shows that the magnitude of negative strains seem to increase with the increase in accumulated forward creep strain. It should be noted that the observation of negative creep is not a transducer problem because this kind of negative creep has been observed when samples were reloaded with the same strain gage or when a new strain gage was attached between the unload and reload sequence.

8.1.2 Discussion of Negative Creep

Negative creep phenomenon has been observed before in nickel based superalloys[1]. Since the lattice parameters of the disordered and the ordered phases are different, it was proposed that stress induced coarsening of the ordered precipitates changed the volume fraction of the two phases. This was thought to cause the observed negative creep in the
superalloys. But this kind of process cannot be expected here because we observe this phenomenon in a single phase alloy. In titanium alloys this has been observed before in two alloys namely, Ti-5Al-2.5Sn (Ti-5-2.5)[2] and Ti-6Al-4V(Ti-6-4)[3]. In the Ti-5-2.5 alloy which is a single phase α titanium alloy, Thompson and Odegard[2] observed negative creep strains of the order of 100 microstrains. Odegard and Thompson[3] observed negative creep in their work on room temperature creep of Ti-6-4. In both studies negative creep was observed when the material was pre-strained at a higher stress level and then reloaded at a lower stress level. Samples of Ti-6-4 were pre-strained by Odegard and Thompson at 95% of its yield strength to various strain levels and subsequently tested them at lower fractions of the yield strength, namely 70%, 80% and 90% of the yield strength. The maximum negative creep strains were observed in the case of the 70% test with the largest pre-strain level and this was around 40 microstrain. They did not observe negative creep at a stress level of 90% of yield strength even for the largest pre-strain levels. But it has to be pointed out that the first data point in their creep curve is after 1 hour and it is possible that negative creep occurred even at this stress level and it was not measured. The magnitude of the negative strain that was observed in this work is similar to what was observed in our work. But one major difference is that in the Ti-6-4 work they observed this phenomenon occurring over hours at lower fractions of yield strength.

From the data shown in table 8.1, it appears as though the forward creep and negative creep processes are occurring in parallel upon reloading. This appears to be a reasonable assumption, because figure 8.3 shows an unload/ reload sequence from an AC sample. Here the initial creep rates are fast enough that one does not observe negative creep but the forward creep rate appears to be close to zero for few tens of seconds upon reloading.
One can imagine that in this case forward and backward creep compensate each other to make the forward creep rate close to zero. This model, then suggests that the duration of observation of negative creep is dependent on the forward creep rate. This is apparent from the data presented in table 8.1. Also, in the work of Odegard and Thompson, the observation of negative creep over a period of hours can be explained by the fact that at the lower stress levels the forward creep rate tends to be very low, hence, one can observe this phenomenon over long times. The other possibility for the absence of negative creep at 90% of the yield strength in Odegard and Thompson's is that the forward creep rate was high enough to mask the occurrence of negative creep.

Odegard and Thompson[3] explained this behavior in terms of internal stresses and suggested that at the lower creep stresses, the internal stresses due to the dislocations generated during pre-straining is larger than the applied stress and causes backflow. Although this is a plausible explanation this does not present the complete picture of this phenomenon. In our case we do not observe backflow after a complete load drop even though the deformation microstructure is dominated by large internal stresses due to pile-ups and negative creep occurs only on subsequent reloading. It appears as though that the material is able to backflow only under a state of high forward stress. It has been shown by Girshick and co-workers[4] that the core of the \( \langle a \rangle \) type screw dislocations in titanium is split both in prism and basal plane. In order for the \( \langle a \rangle \) type screw dislocations to glide on the prism planes the core has to be compacted on the prism plane by a cross slip process. Hence, it is possible that the internal stresses actually spread the core in the basal plane and prevent backflow once the load is dropped below a certain value. On the other hand when we reload the material the forward stress helps in constricting the core of the screw dislocations and the material can now backflow in the regions where the internal stresses are larger than the applied stress. In this picture one
can imagine regions of high internal stresses backflowing upon reload, and other regions of the sample flowing forward where the internal stresses are lower. Hence, the negative flow and forward can be independent processes occurring upon reload and the negative creep can be observed only when the forward flow rates are very low.

8.2 Tension/Compression asymmetry in titanium alloys

8.2.1 Observation of tension/compression asymmetry

Constant strain rate tests and creep tests have been done in tension and compression in two alloys, Ti-6Al-D1 and Ti-6242. Table 8.2 lists the mechanical properties under constant strain rate deformation. There is a small asymmetry in the 0.2% yield strength between tension and compression for both Ti-6Al-D1 and Ti-6242. The material is stronger in compression than in tension. The yield strength difference is 27MPa for Ti-6Al-D1 and 51 MPa for Ti-6242. Figures 8.4 and 8.5 show log-log plots of true strain versus true stress for Ti-6242 and Ti-6Al-D1 respectively. One can see from these plots that the material is stronger in compression and that the flow curves are diverging, indicating different work hardening behavior. The strain hardening exponent, $n$, is smaller in tension than in compression for both alloys. Further, we measured strain rate sensitivity, $m$, from strain rate jump tests in both tension and compression. Both $n$, $m$ along with the strength coefficient $K$ are listed in Table 8.2 for both alloys. The $m$ value is slightly higher in tension compared to compression.

Figure 8.6 shows the creep response of Ti-6Al-D1 at a stress of 716 MPa in both tension and compression. There is a dramatic asymmetry in creep response, much more than what one would expect from the asymmetry in constant strain rate properties. The sample
failed in 40 min in tension at room temperature, even though it was loaded to a stress level lower than its 0.2% yield strength. Primary creep response was followed by steady state like behavior, followed by failure after a tertiary regime. The creep time exponent $a$ (0.6) was very high in the primary regime, indicating very slow exhaustion. This is shown in comparison with the material response in compression in figure 8.7, which is a log-log plot of the same data shown in figure 8.6. In addition, the steady state creep rates are very high ($1.6 \times 10^{-5} \text{ s}^{-1}$) and this is listed in figure 8.6.

Creep tests of Ti-6242 were conducted at two stress levels, 931 MPa and 827.5 MPa in both tension and compression. The linear time versus creep strain plots are shown in figures 8.8 and 8.9. As one can observe from these plots, there is a large asymmetry in the creep behavior even in this alloy. Again, the alloy is much more creep resistant in compression than in tension. Table 8.4 lists the creep strains measured at a particular time between tension and compression for both alloys. Creep times for comparison were chosen to represent the long time creep response. The table also lists the ratio of the creep strain in tension to the creep strain in compression. In Ti-6242 the creep strain accumulation at 931 MPa is 6.7 times more than in compression at $t=550000$ sec. This asymmetry is slightly reduced at the lower stress level of 827.5 MPa. In this case it is 4.9 times higher in tension at $t=4000000$. It should also be noted that we have to wait approximately 7 times longer in time at the lower stress level to obtain this large an asymmetry. This indicates that the divergence between tension and compression is highly stress sensitive. This asymmetry in creep strain is expected to increase with time at the lower stress level because the long time creep exponent was lower in compression. The creep asymmetry is expected to remain the same for the higher stress level in Ti-6242, because in this case the long time creep exponent was observed to be the same for both
tension and compression. Finally, in the case of Ti-6Al-DI, the asymmetry in creep strain is 12.7 even after a short time of 2520 sec.

Using the phenomenological analysis presented in chapter 5, the creep response in tension was predicted for Ti-6242 from the constant strain rate data presented in table 8.2. The calculated creep exponent $A$ and $a$ are listed in table 8.3 for both stress levels. Log-log plot of time versus creep strain is shown in figure 8.10, where the predictions and the experimental data for the two stress levels are presented. The predictions reproduce the stress and time dependencies reasonably well. Clearly, the calculated time exponent, $a=0.3$, is higher than the experimentally observed exponents at long times for both stress levels. But this phenomenological analysis does show that subtle changes in $n$, $m$ and significant changes in strength parameter $K$ can alter the creep behavior dramatically.

8.2.2 Microstructural origin of tension/compression asymmetry

The behavior of Ti-6Al-DI alloy is probably an extreme case. This is a single phase alloy with an as-cast microstructure. Grain/variant size was very large ($\approx 500\mu m$) in this alloy. It is possible that slip transmission between grains is relatively easy in tension in this alloy and also the failure of the sample could be accelerated by presence of voids. Possible explanations for this large asymmetry in creep will be discussed considering the magnitude of asymmetry exhibited by Ti-6242 to be typical. This alloy had an equiaxed microstructure with primary $\alpha$ grains and transformed $\alpha+\beta$ grains. The average grain size of the Ti-6242 alloy is $\approx 20\mu m$.

In the literature, asymmetry in yield strengths between tension and compression have been reported for titanium alloys[5-11]. Small asymmetry in yield strength was reported 269
in Ti-6Al-6V-2Sn and Ti-6-4 by Chait[5] and in Ti-6-4 by Meyer[7]. The yield strength differences were of the order of what was observed in our work. Information about texture of the material is not available in these two works. It is interesting to note that all the alloys were observed to be stronger in compression than in tension, similar to our observations. The works by Jones and Hutchinson[6], Medina Perilla and Gil Sevillano[8], Philippe et al[9], Fundenberger et al[10] and Lecomte et al[11] were all on highly textured polycrystalline Ti-6-4 alloy. Except for the work of Jones and Hutchinson[6], rest of the work were on texture evolution during deformation in Ti-6-4. In all studies the alloy was much stronger in compression than in tension when tested along the orientation in which the stress axis was parallel to the c-axis of the α phase. Jones and Hutchinson[6] proposed that the large asymmetry observed in the yield strength when the alloy was deformed along the c-axis of the alpha phase is due to the nature of motion of <c+a> dislocations. They argued that due to crystal symmetry, +<c+a> and -<c+a> directions are not equivalent. Hence, asymmetry in the mechanical behavior of the hcp-α phase is to be expected when deformed in compression versus tension, if the deformation is accomplished primarily by the motion of <c+a> dislocations (i.e. when deformed along the c-axis). Further, based on a hard sphere model of dislocation motion, they proposed that slip along the +<c+a> direction will be easier than along the -<c+a> direction. Hence, the critical resolved shear stress (CRSS) in compression will be higher than in tension. Another effect of the proposed model, is that there is significant dilatation associated with the <c+a> dislocation motion. Therefore, the hydrostatic stress component of the applied stress tensor should affect dislocation motion.

CRSS for <c+a> motion, measured from deformation along c-axis in highly textured Ti-6-4 from the work of Jones and Hutchinson[6] is shown in table 8.5, along with the data
from the work of Medina Perilla and Gil Sevillano[8]. The table also lists single crystal measurements from the work of Paton et al[12] in a single phase Ti-5wt%Al alloy. Asymmetry in the single crystal data is even higher than those measured from the textured Ti-6-4 data. Single crystal CRSS for \(<c+a>\) slip in compression for a Ti-6.6Al alloy from the work of Paton et al[12] is also listed in table 8.5. CRSS in compression for Ti-6.6 Al and Ti-5Al are comparable and one can expect similar asymmetry in tension for the Ti-6.6 Al alloy, which was not measured by Paton et al. Since the aluminum content in the alpha phase of Ti-6242 is expected to be around 6wt%, one would expect such large asymmetry for \(<c+a>\) dislocation motion between tension and compression in this alloy as well.

Based on the above discussion, one of the first questions that needs to be answered is whether our two alloys exhibit a strong texture and whether we were in fact testing the material along the c-axis of the \(\alpha\) phase. Pole figure measurements were made using both x-rays and orientation imaging microscopy (OIM) on both Ti-6Al-DI and Ti-6242 alloys. Samples were cut from the grip portion of the tensile specimens and many pole figure measurements were made parallel to the tensile stress axis. Additional measurements in other orientations were made using x-rays, in samples cut from grip portion of tensile samples and from the ring forging from which the Ti-6242 samples were machined. It is important to note that both tensile and compression specimens were machined along the same orientation from the as received stock for both alloys. Hence, all the tests were conducted with the stress axis essentially along the same orientation in both compression and tension. Figures 8.11 and 8.12 show the \((0001)\) and \(\{10\bar{1}0\}\) pole figures for Ti-6242 and Ti-6Al-DI respectively. These pole figures were measured using OIM over an area of 16mm\(^2\) and 64mm\(^2\) for Ti-6242 and Ti-6Al-DI respectively. These pole figures show that
there is no preferred orientation along the c-axis and in general there is very little texture
in both alloys. All the x-ray measurements also showed that both alloys do not have any
strong texture. Further, if one compares the asymmetry in yield strengths in our work
with the data presented from the literature for \( <c+a> \) slip in table 8.5, the observed
asymmetry in yield strength is much lower in our case. From the above results, it appears
that the large asymmetry in creep cannot be explained as due to the direct activation of
\( <c+a> \) slip.

A second possibility is that due to residual stored work or due to improper
recrystallization, the asymmetry in creep response is a bauschinger effect. Ti-6Al-D1 is a
cast alloy. Further, Ti-6Al-D1 and Ti-6242 were well annealed high in the \( \alpha \) and \( \alpha+\beta \)
field respectively. Even if there were residual internal stresses in the material, it is not
reasonable to expect the residual stresses to favor tensile deformation for both alloys.
Hence, internal stresses from prior work are expected to play minimal role and cannot
explain the observed behavior.

In order to explain the large asymmetry in creep response between tension and
compression one needs a mechanism by which either slip of the whole sample is easier in
tension, in other words, easier accommodation of elastic and plastic incompatibilities in
tension or slip within the grains are faster in tension, i.e. faster individual or collective
motion of dislocations within a grain. In both cases one would accumulate larger strains
at a given time in tension than in compression.

Deformation in the alpha phase of two phase titanium alloys with 4wt% Al or greater is
expected to be planar. Recently, Lütjering[13] has stated that the \( \alpha/\alpha_2+\alpha \) transus for Ti-
6242 is around 650°C. If one aged the sample inside the two phase region, one would get \(\alpha_2\) precipitates in \(\alpha\) matrix after long time aging. It has been shown by Williams and Lütjering[14] that precipitation of \(\alpha_2\) promotes very planar slip. In our case the Ti-6242 samples underwent a stabilization treatment of 8h anneal at 593°C. One would expect at a minimum, SRO of Ti-Al atoms in the \(\alpha\) phase in our alloy. We have shown in previous chapters, presence of SRO also promotes planar slip. In Ti-6242, the \(\alpha\) phase is present in greater than 90% volume fraction. Further, in this study, Ti-6242 was studied in the equiaxed condition and one would expect the behavior of this alloy to be closer to the behavior of a single phase alloy. Hence, it is reasonable to propose a model for the tension/compression asymmetry in the two phase Ti-6242 alloy based on the understanding of the creep behavior of single phase alloys.

In chapter 6, based on the creep response of Ti-6Al it was shown that there are three regimes of differing exhaustion rates that exist during the primary creep of these alloys. There is an initial regime (I) where the creep exhaustion rate is high (small \(a\) values), then there is second regime (II) where the creep exhaustion rate is lower (large \(a\) value and in some cases an inverse primary has been observed) and there is a long time regime (III) where the exhaustion rates are higher again (smaller \(a\) values). From the creep response of Ti-6Al after various heat treatments and from slip line evolution studies, it was proposed that the initial primary regime represents mainly slip nucleation and propagation within grains which are orientated most favorably for slip. It was also proposed that pile-ups in the slip bands are strongly interacting with the grain boundaries in the first regime. The second regime is thought to occur due to slip transmission/nucleation in grains which are less favorably oriented for slip due to local plastic incompatibilities generated by slip in the `easy' slip grains. This causes a region of
slow exhaustion (or inverse primary in some cases). The final regime represents exhaustion rates when most of the grains are participating in the deformation.

When the material is deformed in tension, it is possible that the slip processes that were discussed above are occurring at a higher rate in tension due to higher mobility of dislocations in tension. In chapter 6, it was shown that deformation in Ti-6Al is heterogeneous. The two main features of the deformation microstructure are that the dislocations are preferentially oriented in the screw direction and there are large internal stress concentrations due to slip propagation through dynamic pile-ups. As discussed in chapter 6, atomistic calculations by many workers[4, 15-21] have shown that the dislocation core structure of $\langle a \rangle$ type screw dislocations in $\alpha$-titanium are spread in both prism and basal planes. This makes the screw orientation the slowest moving line direction of a dislocation loop. If the screw orientation becomes more mobile in tension, then one can in fact expect the creep process to occur at a higher rate. Based on their studies of the core structure of screw dislocations in BCC metals, Vitek and co-workers[22, 23] have shown that due to dislocation core structure there could be non-schmid effects in the deformation behavior. In other words, the CRSS for a particular slip system varies with the orientation of the crystal. They have identified two kinds of non-schmid effects, one was termed intrinsic non-schmid effect, due to the twinning/anti-twining asymmetry in the $\langle 111 \rangle$ slip directions in $\{112\}$ planes. This is a feature unique to BCC metals. One would not expect this behavior for $\langle a \rangle$ slip in titanium alloys due to the crystal symmetry. The second effect was termed extrinsic non-schmid effect. They have shown that shear stresses normal to the burgers can affect the dislocation motion. This is because, if the screw dislocation core structure had edge components, then the shear stress normal to the burgers vector can interact with the edge components and alter
the core structure. Such behavior has been observed in BCC metals. Hence, dislocation motion could be easier in tension or in compression. In the case of titanium alloys, it is possible that such extrinsic non-schmid effects cause the mobility of screw dislocations to be higher in tension. The most recent calculations by Girshick et al.[4] on \(<a>\) type screw dislocation core using a bond order potential showed that the screw dislocations are split on both prism and basal planes in pure \(\alpha\)-titanium and that the core structure does not have significant edge components. This would indicate that such extrinsic non-schmid effects probably will not exist for \(\langle a\rangle\) type screw dislocations in titanium alloys. On the other hand, non-schmid behavior have been reported for prism slip in \(\alpha\)-titanium by Naka et al.[24] and in titanium-aluminum alloys by Sakai and Fine[25]. Naka et al observed that the CRSS for prism slip changed with the orientation of the sample. They suggested that the screw dislocation core structure could be split on the prism plane and symmetrically on two pyramidal planes. Comparing the schmid factor on pyramidal planes for the different orientations, they concluded that the CRSS increased as the schmid factor for one of the pyramidal planes decreased. But the core structure assumed by them for the screw dislocations was not observed in atomistic calculations as discussed above. Sakai and Fine[25] observed that the CRSS for prism slip depended on the orientation of the sample in dilute titanium-aluminum alloys. This was observed when the sample oriented for basal slip (S.F = 0.5) still deformed by prism slip (S.F = 0.22) in dilute Ti-Al alloys. It was also noted by them that as the aluminum content was increased to 5.2 at% Al, the sample oriented for basal slip, slipped on basal planes, but there was extensive cross-slip on prism planes. They also suggested asymmetric core structure to be the cause for this behavior. Figure 8.13 compares the CRSS for prism slip versus basal slip as a function of aluminum content at room temperature; data taken from the work of Paton et al[12]. Table 8.6 shows the ratio of CRSS for basal versus prism slip at room temperature for
dilute titanium-aluminum alloys for aluminum content less than 3 wt% (5.2 at%) taken from the work of Sakai and Fine[26]. From the above data it appears as though prism slip is hardened much more rapidly than basal slip, as the aluminum content is increased. Rosenberg and Nix[27] argued that strengthening due to aluminum in titanium alloys is due to the strong elastic interaction between the edge kinks (of length \( \approx b \)) on screw dislocations with the solutes. Based on this argument, one cannot expect the strengthening of aluminum to be stronger for prism slip compared to basal slip. From x-ray diffraction studies [28, 29], it was concluded that the stacking fault probability on basal planes increased as the aluminum content was increased in binary titanium-aluminum alloys. Recently, Grujicic and Zhou[30], based on atomistic studies, showed that addition of nitrogen to Fe-Ni-Cr austenitic steels altered the core structure of screw dislocations making them less mobile. Hence, it is possible that the core structure of screw dislocations are altered with the addition of aluminum in titanium alloys and it favors larger dissociation on the basal planes. This would suggest that CRSS for basal slip will decrease with increasing aluminum content. It would also increase the CRSS for prism slip due to larger dissociation on the basal planes. But from figure 8.13 it appears as though the CRSS for basal slip remains almost constant with increase in aluminum concentration. One could argue that the expected drop in CRSS is almost compensated by the elastic interaction of solutes in the case of basal slip. The above discussion shows that the screw dislocation core structure in titanium alloys is probably not completely understood, especially in titanium-aluminum alloys. But, based on the results available now, it is not reasonable to expect the \(<a>\) dislocations to behave significantly differently in tension compared to compression.
In an anisotropic polycrystalline material there could be significant elastic compatibility stresses at the grain boundaries as discussed by Hirth[31]. In titanium-aluminum alloys deformation is very planar and proceeds through large dislocation pile-ups. Hence, one can expect significant interaction between the dislocations in the slip bands and grain boundaries in these alloys. The large stress concentrations generated by the pile-ups at grain boundaries and the elastic incompatibility stresses can be relieved by either of three mechanisms[32]. Firstly, slip transmission can occur through the grain boundary. This process entails supplying energy for the creation of a grain boundary step and for the residual dislocations left in the boundary. A second possibility is the operation of a grain boundary dislocation as a dislocation source and the third possibility is the activation of a Frank-Read source near the boundary in the neighboring grain.

Slip transmission across a general grain boundary in α-titanium has been studied recently[33, 34]. Based on their work on in-situ tensile deformation studies in a TEM, Shirokoff et al[33] showed that slip transmission can in fact occur in α-titanium at a general grain boundary. Dislocations were observed to pile-up at a grain boundary. They were accommodated in the grain boundary until the stresses became large enough for slip transmission to occur. It was observed that the burgers vector with minimum rotation, maximum shear stress and leaving the smallest residual burgers vector content at the grain boundary was chosen in the neighboring grain during such a slip transmission event. Further, they noted that the residual burgers vector content decomposed into lattice dislocations and moved into the grain into which slip transmission occurred. It is interesting to note that one of the decomposition products of the residual burgers vector were \(<c+a>\) lattice dislocations. In a general case one would expect the residual dislocations at the boundary to have a \(<c>\) component when such slip transmission events
occur. And it is reasonable to expect the residual dislocations to decompose into perfect lattice dislocations, if such a reaction is possible, to minimize the elastic energy at the boundary. Such a slip transmission process can continue to occur at a particular location only if the residual dislocation content is removed or reduced. Otherwise, the residual dislocation content at the grain boundary will oppose further slip transmission. It was discussed before that the CRSS for \(<c+a>\) slip is much higher in compression than in tension. Hence, in principle one would expect the slip transmission process to be much more difficult in compression than in tension, if such a process involves the formation and glide of \(<c+a>\) dislocations. Kehagias and co-workers[34] studied slip transmission in rolled \(\alpha\)-titanium after deformation. Even in this post-mortem study they observed slip transmission events across a general grain boundary. But, they did not observe any decomposition of residual burgers vector near the boundary.

In a general polycrystal it is not clear at this point how important are such slip transmission events. Recently, in polycrystalline Nb-Ti-Al intermetallic alloys large ductility has been observed at room temperature, even though the macroscopic stress strain curve exhibits work softening[35]. The dislocation microstructure consists of large pile-ups of screw dislocations, similar to titanium alloys. It was suggested that easy slip transmission across grain boundaries was the reason for the observed large ductilities even though the alloy exhibited work softening. This study would suggest that slip transmission could be an important process in polycrystalline plasticity.

Grain boundaries can act as dislocation sources[31, 36]. In titanium alloys, Thompson and Odegard[2] reported that \(\alpha'\) martensite boundaries acted as dislocation sources in a Ti-5Al-2.5 Sn alloy. Odegard and Thompson[3] observed a general grain boundary acting...
as dislocation sources in Ti-6-4. We have observed grain boundaries acting as dislocation sources in Ti-6Al alloys as shown in chapter 6. Hence, grain boundary sources can be activated to relieve stresses at grain boundaries. Slip line observations in copper, 70-30 brass and 99.9 % iron has shown that in addition to main slip systems, secondary slip systems were activated near grain boundaries to accommodate inhomogenieties across the boundary[37]. Further, these secondary slip lines were observed to move into the grain[37]. Hence, dislocations generated to relieve local incompatibility stresses can glide into the grain interior and contribute strain.

Based on the above discussion, it is clear there could be many microscopic processes of slip accommodation. In general these would involve nucleating secondary slip systems in addition to the main slip systems. One can expect in polycrystalline titanium alloys that slip continuity across grain boundaries would require significant operation of <c+a> dislocations. Figure 8.14 shows interaction of lattice dislocations at a grain boundary in a AC sample deformed in compression. One can observe significant <c+a> slip activity near the grain boundary. These dislocations have been observed to move into the grain contributing to the overall creep strain. From the behavior of <c+a> dislocations in tension and compression, it would be appear that such slip accommodation processes will be much easier in tension than in compression. Note that the work of Paton et al[12] indicates the CRSS of <c+a> dislocations to be much higher than <a> dislocation. Thus, the presence of <c+a> dislocations such as this is proof that large compatibility stresses exist.

The primary creep data in tension and compression shown in figures 8.8 and 8.9 were re-plotted in log-log axes to identify the three regimes of exhaustion behavior in Ti-6242 as
shown in figures 8.15 and 8.16. From these log-log plots it appears that the three regimes of transient response do exist for Ti-6242 in both compression and tension. This is especially clear at the lower stress level. The initial creep rates and the $a$ values in the three regimes are compared for the four tests in table 8.7. The initial creep rates appear to be higher in tension than in compression at the lower stress level (measured between $t = 10s$ to $50s$). The most important characteristic of the creep curves between tension and compression, is that the creep response starts out similar, but the curves start to diverge with time. The regime of lower exhaustion rate is longer and the $a_{\text{II}}$ value in this regime is higher in tension than in compression (especially at the lower stress level). The long time exponent ($a_{\text{III}}$) is similar in both tension and compression at the higher stress level, but is slightly different at the lower stress level.

Based on the discussion of slip accommodation at grain boundaries, one would expect the higher $a$ regime to occur early and have longer duration if the accommodation processes were easier. This is because, in regime I during the initial primary, interaction of grain boundaries with rapidly formed pile-ups is the only significant hardening process. Grain boundaries can be effective barriers for further slip, if slip accommodation in the neighboring grains is difficult. If one assumes that such processes involve nucleation and motion of $<c+a>$ dislocations, then in compression, it would require larger stress concentration build up at the boundaries before slip accommodation occurs. This would imply smaller ‘$a$’ values in compression, indicating stronger exhaustion. Eventually, slip accommodation will occur with time and leads to the higher ‘$a$’ regime. In comparison, one would require less stress concentrations in tension. Hence, the ‘$a$’ value in the initial regime will be higher, indicating slower exhaustion. Further, with continued slip more strain can be accumulated per unit time in tension because of easy slip propagation.
through many grains. The larger ‘a’ regime will occur for longer time in tension. This is what is observed in tension compared to the creep response in compression. At this point it is not clear whether the \(<a>\) dislocation mobility is higher in tension than in compression. Hence, the large asymmetry in creep response could be primarily due to the ease of slip accommodation in tension due to easier \(<c+a>\) dislocation motion.

It was discussed before, that based on the hard sphere model of \(<c+a>\) motion proposed by Jones and Hutchinson[6], one would expect the hydrostatic component of the stress tensor to have significant effect on the \(<c+a>\) dislocation motion. This is due to the dilatation associated with the motion of such dislocations. Compression tests were conducted with and without superimposed pressure in an AC sample. The data from these tests are listed in table 8.8, showing that there is in fact little effect of the hydrostatic pressure in the 0.2% yield strength. If similar tests were conducted in tension, there could be a difference in the yield strength due to the superimposed pressure. Because it is reasonable to expect \(<c+a>\) dislocations to play a role in the yield process itself due to both plastic and elastic compatibility stresses.

### 8.3 Room temperature recovery

Room temperature recovery has been observed in both Ti-6242 and Ti-6Al alloys. After creep testing, if the sample was kept at room temperature for a few months and then reloaded, it was observed that sample exhibited a creep response similar to well annealed material being deformed for the first time. In other words, the creep behavior after the reload indicates little effect of prior work hardening that has occurred during the first loading. Figures 8.17 and 8.18 shows creep time versus strain plots of AC and Ti-6242 samples respectively. These samples were loaded and crept for a few days.
unloaded and left at room temperature for a few months (this is indicated in the curves for both alloys). Then the samples were reloaded to the same stress level as before. The material response is dramatically different after the reload. It appears as if the material has been loaded in the as annealed condition and initial creep rates are a few orders of magnitude higher than the rates observed before unload. These values are tabulated in table 8.9.

When Ti-6Al-DI samples were unloaded and reloaded after 20-24h. The material response was a continuation of the creep behavior before unload. A Ti-6242 sample was unloaded and reloaded after 200h, even in this case the material response was a continuation of the behavior before the unload. The strain rates before unload and after reload from these short time tests are also listed in table 8.9. It can be seen that even after a short waiting time there is a difference in the initial creep rates, but the material response follows the pre-unload behavior after an initial transient period. An IWQ sample was unloaded and reloaded after 6 months and 10 days. In this case also the initial rates were higher after the reload, but the creep behavior followed the pre-unload behavior after an initial transient period, similar to the short time reload samples. This is shown in figures 8.19 and 8.20.

It was shown in the chapter 6 that AC sample deforms in a planar fashion and the microstructure consists of large pile-ups. The IWQ sample deforms more or less in a homogeneous fashion. Therefore, there is a significant difference in the microstructural condition between the two samples. In the case of AC sample there are large internal stress concentrations due to the pile-ups, both in the interior of the grains and at grain boundaries. Such planar deformation is shown in figure 8.21 of an AC sample. In the
FCC-SRO alloys, formation of multipoles have been observed after deformation. Such low energy structures are not formed in the case of titanium alloys when deformation is planar. Where as, in the case of IWQ sample there are no such large stress concentrations especially at the grain boundaries.

Finally, sample dimensions were measured many months after deformation. This would indicate a change in the dimensions if there was any forward flow in the unloaded condition due to the large internal stresses in the alloys exhibiting planar slip. There was no measurable change in the dimensions to indicate that there was any forward flow in the unloaded conditions. The present results indicate that the internal stress concentrations due to the pile-ups could be causing the observed room temperature recovery. Relief of local stress concentrations via creep processes, for example, generation of fresh dislocation sources can occur. These sources upon external reloading, are ready to produce larger strain rates than expected based on previous loading transient. But the exact mechanism for this behavior is not known at the present time.

8.4 Summary

(a) Negative creep has been observed in titanium alloys, where the sample expands against the applied stress when reloaded after some pre-deformation. This has been tentatively attributed to the unique core structure of $\langle a \rangle$ type screw dislocations.

(b) A large asymmetry in creep response of Ti-6Al-D1 and Ti-6242 has been observed between tension and compression at room temperature. A smaller asymmetry has been observed between tension and compression in constant strain rate deformation. Under both deformation conditions the material is stronger in compression. It was shown that
there is no pronounced texture in both alloys that were tested to suggest that the asymmetry is due to direct activation of \(<c+a>\) dislocations. From the present understanding of dislocation core structures in titanium alloys, it is not reasonable to assume the \(<a>\) dislocation mobility is higher in tension compared to compression. But it was shown that clear understanding of the influence of aluminum content on the dislocation core structures is not known presently. Hence, such mobility arguments might still be valid. Based on slip accommodation processes across grain boundaries in a polycrystalline material, it is suggested that such processes will require activation and motion of \(<c+a>\) dislocations. Slip accommodation would be easier in tension than in compression because of easier activation of \(<c+a>\) slip in tension. It is proposed that such processes could be the reason behind the observed large asymmetry between tension and compression.

c) Room temperature recovery has been observed in Ti-6242 and in an AC sample. Samples deformed after a pre-deformation many months earlier exhibit creep behavior similar to well annealed material. But such recovery is not observed if the samples are reloaded within 200h. In all cases the samples were stored at room temperature between pre-deformation and subsequent deformation. Measurement of sample dimensions after many months, does not show any indication of forward flow occurring in the unloaded condition. An IWQ sample does not exhibit such room temperature recovery. Based on the microstructural differences between IWQ and AC sample, it is proposed that the observed behavior is due to the influence of the large internal stresses present in the alloys deforming in a planar fashion.
References


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Figure 8.1: No observable back-flow in a Ti-6Al-DI sample after a complete load drop. The sample was crept at a stress of 716MPa to a creep strain of 1.6%.

Figure 8.2: Total strain versus total time plot showing the occurrence of negative creep after unloading and subsequent reloading of a Ti-6Al-DI sample.
Figure 8.3: No backward or forward flow was observed for the first few seconds in a AC sample after reloading to the original stress. In this sample the forward flow rate immediately after this no flow regime was observed to be $7.4 \times 10^{-7} \text{s}^{-1}$.

Figure 8.4: Log-log plot of true strain versus true stress for Ti-6242 measured in tension and compression. The sample is stronger in compression and has a higher work hardening exponent in compression. The material properties are compared in table 8.2.
Figure 8.5: Constant strain rate response of a Ti-6Al-DI alloy in tension and compression is shown here. Log-log plot of true strain versus true stress shows that the alloy is stronger in compression than in tension and has a higher work hardening exponent in compression compared to tension. The material properties are compared in table 8.2.

Figure 8.6: Linear creep time versus creep strain plot shows the large asymmetry in the creep response between tension and compression in a Ti-6Al-DI alloy. The tensile sample failed in 40 min at a stress level below the yield stress at room temperature. The sample exhibited steady state creep and the creep rate is shown in the plot.
Figure 8.7: Here the log-log plot of the same data shown in the previous figure is presented. In tension, the sample exhibits a large creep time exponent (\(a=0.6\)) in the primary regime before steady state and tertiary regimes are reached.

Figure 8.8: Creep response of Ti-6242 in tension and compression at the same stress level of 931 MPa. The yield strength of the material in tension is 931 MPa. There is a large asymmetry in creep response between tension and compression.
Figure 8.9: Comparison of creep response between tension and compression in Ti-6242. Both the tests were done at the same stress level of 827.5 MPa. This stress level represents 88% of the yield strength of the material in tension. There is still a large asymmetry between tension and compression even at this lower stress level.

Figure 8.10: Log-log plot of creep time versus creep strain of Ti-6242 tested at two stress levels in tension. The predicted creep curves were calculated from the measured hollomon parameters listed in table 8.3 using the analysis presented in chapter 5. Stress dependence and larger strain accumulation in tension are reproduced reasonably well.
Figure 8.11: (a) and (b) Pole figures of Ti-6242 taken from the grip section of the deformed sample, along the stress axis of the sample. The sample does not show significant (0001) texture and in general exhibits minimal texture.
Figure 8.12: (a) and (b) Pole figures of Ti-6Al-DI taken from the grip section of the deformed sample, along the stress axis of the sample. The sample does not show strong (0001) texture and in general minimal texture.
Figure 8.13: CRSS for basal and prism slip as a function of aluminum content at 300K. taken from the work of Paton et al [12]. Aluminum seems to strengthen prism slip more rapidly than basal slip.

Figure 8.14: Activation of \(<c+a>\) slip near a grain boundary in an AC sample deformed in compression. These \(<c+a>\) dislocations were observed to move into the grain interior. The \(<a>\) slip bands and the \(<c+a>\) bands have been labelled in the image.
Figure 8.15: Log-log plot of creep time versus creep strain of Ti-6242 in tension and compression. This is the data shown in figure 8.8 but plotted in the log-log scale. It shows that there are three regimes in the creep transient. (see text for details).

Figure 8.16: The data shown in figure 8.9 is re-plotted here in log axes. At this lower stress level the three regimes of creep exhaustion can be clearly seen. Also regime II exhibits much slower exhaustion and occurs over longer times in tension compared to compression.
At $\Delta t = 4$ months $\pm 14$ days
Stress = 552 MPa

Creep rate before unload = $7.4 \times 10^{-9}$ s$^{-1}$
Creep rate upon reload = $7.4 \times 10^{-7}$ s$^{-1}$

Figure 8.17: Creep time versus creep strain plot in an AC sample showing significant strains after the sample is reloaded after 4 months. This indicates significant recovery at room temperature.

At $\Delta t = 11$ months
Stress = 827.5 MPa

Creep rate before unload = $3.0 \times 10^{-11}$ s$^{-1}$
Creep rate upon reload = $1.8 \times 10^{-7}$ s$^{-1}$

Figure 8.18: Linear creep time versus creep strain plot showing room temperature recovery effect in the commercial Ti-6242 alloy.
Figure 8.19: Unload/reload sequence in a IWQ sample. There is an initial transient response upon reload, where the creep rates are higher than the rates before unload. But the creep behavior is essentially a continuation of the response before unload.

Figure 8.20: Same data as in figure 8.19 plotted in log-log axes. This illustrates that the creep response shows minimal change after the reload.
Figure 8.21: Extremely planar deformation is observed in an AC sample. There are large internal stresses due to the dislocation pile-ups. Unlike the FCC-SRO alloys where large multipole configurations have been observed, such low energy configurations are not observed in titanium alloys.
Table 8.1: Magnitude and duration of observed negative creep response in various alloys. The table also lists the creep strain before the unload/reload sequence and the stresses before unload and after reload. See text for detail.

<table>
<thead>
<tr>
<th>Material</th>
<th>Creep stress (MPa)</th>
<th>Creep strain before</th>
<th>Negative creep</th>
<th>Creep rate after</th>
<th>Duration of</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>/Reload Stress(MPa)</td>
<td>unload/reload</td>
<td>strain (µε)</td>
<td>reload (s⁻¹)</td>
<td>response (sec)</td>
</tr>
<tr>
<td>Ti-6Al D1</td>
<td>716/716</td>
<td>0.002</td>
<td>30</td>
<td>1.0 x 10⁶</td>
<td>23</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.0023</td>
<td>66</td>
<td>1.0 x 10⁶</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.0057</td>
<td>120</td>
<td>4.5 x 10⁷</td>
<td>100</td>
</tr>
<tr>
<td>Ti-6242</td>
<td>827.5/827.5</td>
<td>0.0013n</td>
<td>11</td>
<td>2 x 10⁻⁹</td>
<td>440</td>
</tr>
<tr>
<td></td>
<td>827.5/745</td>
<td>0.0017</td>
<td>24</td>
<td>-</td>
<td>350</td>
</tr>
<tr>
<td></td>
<td>827.5/827.5</td>
<td>0.0017</td>
<td>23</td>
<td>1.8 x 10⁻⁷</td>
<td>30</td>
</tr>
</tbody>
</table>

n- new strain gage was attached to the sample face.
<table>
<thead>
<tr>
<th>Material</th>
<th>Test type</th>
<th>0.2% yield strength (MPa)</th>
<th>K (MPa)</th>
<th>n</th>
<th>m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6242</td>
<td>Tension</td>
<td>931</td>
<td>1216</td>
<td>0.03</td>
<td>0.013</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>982</td>
<td>1430</td>
<td>0.05</td>
<td>0.01</td>
</tr>
<tr>
<td>Ti-6Al-DI</td>
<td>Tension</td>
<td>745</td>
<td>-</td>
<td>0.025</td>
<td>0.0195</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>772</td>
<td>1143</td>
<td>0.04</td>
<td>0.185</td>
</tr>
</tbody>
</table>

Table 8.2. Comparison of material properties between tension and compression measured in constant strain rate tests for both Ti-6242 and Ti-6Al-DI alloys.

<table>
<thead>
<tr>
<th>Material</th>
<th>Stress (MPa)</th>
<th>A</th>
<th>a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6242</td>
<td>931</td>
<td>0.0029</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>827.5</td>
<td>0.000188</td>
<td>0.3</td>
</tr>
</tbody>
</table>

Table 8.3: Calculated creep parameters A and a for Ti-6242 in tension. The parameters were calculated using the material parameters listed in table 8.2 using the analysis presented in chapter 5.
<table>
<thead>
<tr>
<th>Material</th>
<th>Stress (MPa)</th>
<th>Test type</th>
<th>Creep strain (%) (at t-sec)</th>
<th>$\varepsilon_{\text{creep,Ten}}$</th>
<th>$\varepsilon_{\text{creep,Comp}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6242</td>
<td>931</td>
<td>Tension</td>
<td>6.4 (550,000)</td>
<td>6.7</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Compression</td>
<td>0.95 (550,000)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>827.5</td>
<td>Tension</td>
<td>0.83 (4000,000)</td>
<td>4.9</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Compression</td>
<td>0.17 (4000,000)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ti-6Al-DI</td>
<td>716</td>
<td>Tension*</td>
<td>4.7 (2520)</td>
<td>12.7</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Compression</td>
<td>0.37 (2520)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

+-sample failed in tension in 40 min at room temperature.

Table 8.4: The asymmetry in creep properties are listed in the table for both Ti-6242 and Ti-6Al-DI alloys. For the Ti-6242 alloy there is still a large asymmetry in creep resistance between tension and compression even at the lower stress level.
<table>
<thead>
<tr>
<th>Material</th>
<th>Test type</th>
<th>CRSS (MPa)</th>
<th>0.5% Yield Strength (MPa)</th>
<th>CRSS\textsubscript{comp} / CRSS\textsubscript{tension}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-5Al[12]</td>
<td>Tension</td>
<td>300</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>740</td>
<td>-</td>
<td>2.47</td>
</tr>
<tr>
<td>Ti-6Al-4V[6]</td>
<td>Tension</td>
<td>441</td>
<td>1090</td>
<td>1.43</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>631</td>
<td>1557</td>
<td></td>
</tr>
<tr>
<td>Ti-6Al-4V[8]</td>
<td>Tension</td>
<td>494</td>
<td>-</td>
<td>1.24</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>612</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Ti 6.6Al[12]</td>
<td>Tension</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>790</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

Table 8.5: CRSS for \(<c+a>\) slip in tension and compression taken from the literature. There is a large asymmetry in the CRSS and it is higher in compression. The data presented for Ti-6Al-4V is measured from a highly textured alloy and the data for Ti-5Al is from single crystal deformation.

<table>
<thead>
<tr>
<th>Al content (at%)</th>
<th>(\tau_{\text{basal}}/\tau_{\text{prism}})</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.44</td>
<td>5.34</td>
</tr>
<tr>
<td>0.87</td>
<td>6.85</td>
</tr>
<tr>
<td>1.2</td>
<td>6.33</td>
</tr>
<tr>
<td>2.11</td>
<td>4.43</td>
</tr>
<tr>
<td>5.2</td>
<td>2.43</td>
</tr>
</tbody>
</table>

Table 8.6: Ratio of CRSS for basal slip versus prism slip as function of aluminum content at 298K taken from the work of Sakai and Fine[26]. It shows that the CRSS ratio decreases with increasing aluminum content.
<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>Test type</th>
<th>Creep rate (s(^{-1}))-initial</th>
<th>(a_i)</th>
<th>(a_{II})</th>
<th>(a_{III})</th>
</tr>
</thead>
<tbody>
<tr>
<td>931</td>
<td>Tension</td>
<td>-</td>
<td>-</td>
<td>0.33</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>9x10(^{-5})</td>
<td>0.2</td>
<td>0.3</td>
<td>0.14</td>
</tr>
<tr>
<td>827.5</td>
<td>Tension</td>
<td>1x10(^{-6})</td>
<td>0.12</td>
<td>0.45</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>3.3x10(^{-7})</td>
<td>0.03</td>
<td>0.29</td>
<td>0.14</td>
</tr>
</tbody>
</table>

Table 8.7: The initial creep rates and the creep time exponents in the three regimes between tension and compression are compared here. The data presented is for Ti-6242.

<table>
<thead>
<tr>
<th>Superimposed Pressure (MPa)</th>
<th>0.2% yield strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>atmospheric</td>
<td>612</td>
</tr>
<tr>
<td>450</td>
<td>613</td>
</tr>
<tr>
<td>515</td>
<td>622</td>
</tr>
</tbody>
</table>

Table 8.8: Effect of hydrostatic pressure on compressive yield strength in an AC sample. The data shows that there is little effect on the yield strength due to the superimposed hydrostatic pressure.
### Table 8.9: Creep Stress

<table>
<thead>
<tr>
<th>Material</th>
<th>Creep Stress (MPa)</th>
<th>Time between unload/reload</th>
<th>Creep rate before unload ($s^{-1}$)</th>
<th>Creep rate after reload ($s^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-DI</td>
<td>716</td>
<td>20 h</td>
<td>$3.5 \times 10^{-6}$</td>
<td>$1 \times 10^{-6}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>23.5 h</td>
<td>$7.3 \times 10^{-7}$</td>
<td>$1 \times 10^{-6}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 h</td>
<td>$2.4 \times 10^{-7}$</td>
<td>$4.5 \times 10^{-7}$</td>
</tr>
<tr>
<td>Ti-6Al-AC</td>
<td>552</td>
<td>4 mon + 14 d</td>
<td>$7.4 \times 10^{-9}$</td>
<td>$7.4 \times 10^{-7}$</td>
</tr>
<tr>
<td>Ti-6Al-IWQ</td>
<td>552</td>
<td>6 mon + 10 d</td>
<td>$1.3 \times 10^{-8}$</td>
<td>$7 \times 10^{-8}$</td>
</tr>
<tr>
<td>Ti-6242</td>
<td>827.5</td>
<td>200 h</td>
<td>$3.8 \times 10^{-10}$</td>
<td>$2 \times 10^{-9}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>11 mon</td>
<td>$3 \times 10^{-11}$</td>
<td>$1.8 \times 10^{-7}$</td>
</tr>
</tbody>
</table>

Table 8.9: The time between unload/reload and the creep rates before unload and after reload are listed. Much higher creep rates are observed after the reload if the recovery time is of the order of months. The IWQ sample (and Ti-6242 after 200h-waiting time) does show higher creep rates upon reload, but the strain rates come down to the previous levels after a short transient period.
CHAPTER 9

SUMMARY

9.1 Phenomenological analysis of room temperature creep

Creep and constant strain rate deformation was correlated in chapter 5. It was shown that constant strain rate deformation of titanium alloys, studied in this work can be represented by the strain-rate sensitive Hollomon law (equation 5.4). Creep behavior of titanium alloys can be represented by a power in law time (equation 5.1). In chapter 5 a relationship was derived between these two laws (equation 5.8 and 5.9) and the creep behavior of Ti-6Al and Ti-6242 was predicted reasonably well, including the stress dependence. The prediction was especially good for the commercially important Ti-6242 alloy. Such a correlation between constant strain rate deformation and creep deformation also provided a basis for comparing the creep behavior of titanium alloys with other conventional metals and alloys. Based on this comparison, it was shown that the time exponent $a$ for titanium alloys is abnormally high. In fact titanium alloys appear to exhibit creep behavior similar to the behavior of nickel and Cr-Mo steels at much larger homologous temperatures. Finally, phenomenological analysis showed that the strain rate sensitivity, $m$, of titanium alloys is not abnormally high compared to other metals and alloys. But the strain hardening exponent, $n$, of titanium alloys is abnormally low.
Therefore, room temperature creep of titanium alloys can be rationalized as due to this combination of very low $n$ values and moderate $m$ values.

9.2 Characterization of SRO and its effect on room temperature creep

Neutron diffraction was used to characterize SRO in a Ti-6Al alloy. These results were presented in chapter 4. Direct diffraction evidence for the presence of SRO in titanium alloys was presented for the first time. The position of the diffuse peak indicated that the SRO is related to the ordered $\alpha_2$ structure and can be thought of as a precursor to the ordered $\text{Ti}_3\text{Al}$ phase. It was shown that there is significant SRO after a conventional treatment like an air cool. After an IWQ treatment the diffuse peak was very weak indicating the SRO state to be weak. When a sample was aged subsequently at 500°C for 1h after an IWQ treatment, the magnitude of the peak increased significantly (figure 4.4). This indicated that the SRO state can be altered by heat treatment.

Presence of SRO influenced the deformation behavior by promoting planar slip. During deformation the destruction of SRO by the leading dislocation(s) effectively creates a diffuse APB of energy $\gamma_{\text{SRO}}$. Since there is only short-range order, the trailing dislocations in the slip plane cannot heal the diffuse APB. Hence, the leading dislocation(s) experiences a friction due to the creation of $\gamma_{\text{SRO}}$, whereas the trailing dislocations do not experience any friction because they do not alter the SRO state. This leads to a glide plane softening effect causing planar deformation in the presence of SRO. Neutron diffraction results indicated that the SRO state is the weakest after an IWQ treatment. This was also supported by room temperature creep studies. Deformation was observed to be homogenous after the IWQ treatment (figure 6.17). SRO is weak enough in this condition that the destruction of SRO did not provide a glide plane softening effect to
promote planar slip. In the case of IWQ-650C-24h sample and SQ-650C-24h sample, neutron diffraction results indicated that there is little difference in the SRO state between the two samples. This is reasonable because the samples were aged above the $\alpha/\alpha_2+\alpha$ transus and the temperatures were high enough that the SRO state was similar between the two samples. Creep deformation results also indicated that this might be the case because the creep transients were similar for both samples.

Such direct correlation between the neutron diffraction results and creep behavior was not always possible. In the case of AC and AC-400C-5h samples, there was a significant difference in the magnitude of diffuse peak with the latter sample showing a stronger peak. The overall creep transient was very similar between the two samples, but there was a significant difference in the initial response of the two samples. In the case of SQ-600C-24h and SQ-600C-24h-400C-5h samples, neutron diffraction results indicated that there was little difference in the SRO state between the two samples. But the creep response is very different between the two samples, with the SQ-600C-24h-400C-5h showing much higher creep resistance (figure 6.7). Further, it should be noted that differences in the magnitude of diffuse peak were small for all the ageing treatments studied. Thus, if SRO is solely responsible for the differences observed in the creep transients, then this is due to quite subtle ordering effects. In future work, the Warren-Cowley parameters will be determined which may provide insight into such subtle effects.

Creep deformation after different ageing treatments to alter the SRO state produced several types of creep transients. Significant differences in the creep transients were observed in the early time regime, while the creep response was very similar among the
samples at long times. Essentially, three different types of creep transients were observed. IWQ and IWQ+aged samples exhibited three regimes with different exhaustion rates: an initial regime of high exhaustion rate, a second regime of lower exhaustion rate and a final regime of higher exhaustion rate. A second set of samples exhibited only two regimes of exhaustion rates (e.g. AC samples). A regime of low exhaustion rate and a final regime of higher exhaustion rate. Some of these samples exhibited an initial "incubation" period for a few seconds where the creep rates were very low ($\approx 10^{-9} - 10^{-8}$ s$^{-1}$). Finally, SQ-600C-24h-400C-5h and SQ-400C-5h samples exhibited very complex primary transients. They exhibited an initial "incubation" period, after which an actual inverse primary transient (with increasing creep rates and $\alpha$ values greater than 1) was observed for a short time. This was followed by a normal primary (with relatively large $\alpha$ values, see table 6.4) and then a second inverse primary where the creep rates increased for thousands of seconds, followed finally by a normal transient. As the above discussion shows, at short times, there can be a significant deviation from the power law in time (equation 5.1) which has been reported, previously to be the characteristic form of low temperature creep in titanium alloys.

Slip line evolution study provided some insight into the deviation from the power law behavior at early times. The initial regime of rapid exhaustion was identified as due to strong interaction of slip bands activated with grain boundaries in favorably oriented grains. The second regime of slower exhaustion may be due to transmission of slip to neighboring grains. Since slip is propagating in less favorably oriented grains, in which the propagation of slip takes appreciable time, this causes a regime of low exhaustion. At longer times possible interaction between parallel slip bands and intersection of slip bands were identified as leading to higher exhaustion rates.
Microstructural studies revealed that in the Ti-6Al alloys used for studying the effect of SRO on creep deformation, there are no obvious dislocations present inside the grain. Dislocations sources have to be present at grain boundaries and such sources have been identified in these samples. Based on TEM observations and slip line evolution studies, a dislocation model was proposed to explain the features of the observed primary transients. Upon loading the dislocation sources in the favorably oriented grains are activated. Since the edge/mixed segments are much more mobile than the screw segments, beyond a critical separation between the screw dipole, the edge segment will run to the other end of the grain. It is important to note that for further dislocation generation, the screw segments have to move apart a larger distance until the back stress on the source is lowered, so that a positive effective stress acts on the source. This process could be difficult initially because the leading dislocations are experiencing a friction stress due to the presence of SRO. As more dislocations are produced by the source, the back stress on the source will become larger; the screw dipoles must move apart to large distances to provide relief of this back stress. Therefore, it is suggested that if the SRO is relatively homogenous and strong, the initial source dynamics can be very slow. Significant macroscopic strains can be measured only when motion of screw dislocations occurs. The observation of an “incubation” period could be due to such slow source dynamics. Slip line evolution studies showed that there is a sequence of slip processes that occur during creep. As mentioned above, initially favorably oriented grains deform, and slip is transmitted to neighboring grains with time. At long times, essentially many grains in the sample are participating in the deformation. This suggests that initial transients upon loading are probably determined mainly by the deformation characteristics of favorably oriented grains, while the transient in the second regime is
determined by the deformation characteristics of both favorably oriented grains and those neighboring grains to which slip has been transmitted. During the final regime, deformation is occurring probably in the whole sample and the creep dynamics are determined by interaction of parallel slip bands, intersection of slip bands and interaction of slip bands with grain boundaries. Such a model implies that if the initial creep rates are low and comparable to creep rates occurring during the regime of low exhaustion, there will essentially be only one regime of very low exhaustion rate. In the extreme, an inverse primary may be observed due to slip processes in the initial grains, and then a normal primary due to interaction of slip bands with the grain boundaries. Nucleation of slip in the neighboring grains will occur only if there is a sufficient stress concentration at the grain boundary. If the dynamics of deformation in favorable grains are slow, then naturally the slip transmission process will be sluggish as well and we can capture the effect of the source dynamics in favorable grains. Once slip is nucleated in the neighboring grains, there will be acceleration in creep rates because the initial rates are so low. It is suggested that such processes are occurring in the SQ-400C-5h and SQ-600C-24h-400C-5h samples leading to complex primary transients.

9.3 Measurement of $\gamma_{sro}$

The diffuse APB energy created due to destruction of SRO, $\gamma_{sro}$, has been calculated in fcc alloys using dislocation structures as well as theoretically from measured SRO states. Such measurements have not been performed in hcp or titanium alloys before. In principle, such a quantity should be different for faults on basal, prism and pyramidal planes. We have measured $\gamma_{sro}$ on prism planes in an AC, SQ-600C-24h-400C-5h and a IWQ-350C-100h sample using the observed “hair-pin” structures. These structures are essentially leading pairs of edge and screw segments elongated in the screw direction.
From the separation of the screw dipoles, $\gamma_2^{SRO}$, was evaluated for the three samples. These were estimated to be between 25 to 48 mJ/m². These values are comparable to the estimates in fcc alloys. Further, the value of $\gamma_1^{SRO} - \gamma_2^{SRO}$ was estimated to be about 6 mJ/m² in a SQ-600C-24h-400C-5h sample. In addition, a velocity argument complimentary to those in fcc alloys was used to evaluate $\gamma_{SRO}$ from the stress concentration at the head of dynamic pile-ups. These were evaluated in a SQ-600C-24h-400C-5h sample to be between 17 to 43 mJ/m². Again these estimates are comparable to those estimated from “hair-pin” structures and also with those estimated in fcc alloys. In summary, strengthening due to SRO in titanium alloys has been quantitatively estimated for the first time. Although neutron diffraction studies did not show significant differences in the diffuse peak between the aged samples, TEM studies revealed that there is stronger pairing of the lead dislocations in the SQ-600C-24h-400C-5h sample compared to all the others. This sample also exhibited the most creep resistance particularly, implying that there could be subtle differences in the SRO state which are not directly revealed in the “raw” diffraction patterns.

9.4 Microstructural rationale for low $n$ values

The phenomenological analysis discussed in chapter 5 indicated that titanium alloys have low work-hardening exponent, $n$. It was discussed in chapter 6 that the presence of SRO lowers work-hardening rates in fcc alloys due to promotion of planar slip. However, it was shown that work-hardening characteristics of fcc alloys remain much better than those exhibited by titanium alloys. Promotion of planar slip due to SRO cannot completely explain the low work-hardening characteristics of titanium alloys. As discussed in chapter 6, several microstructural reasons can also be identified for this abnormal behavior. Unlike the fcc alloys, there is no multipole formation in titanium
alloys. Due to planar slip elastic interaction and intersections between slip bands is decreased. Such interactions between parallel slip bands have been identified in chapter 6. But even when slip band intersections occur, in many cases the slip bands appear to pass through each with only minor difficulty. They tend to remain planar even after such an intersection process. Fundamentally, co-planarity of the $<a>$ type slip vectors could be the reason behind such a behavior. Some observations were made at the optical level that indicated that slip band intersections do provide some work-hardening. At the present time details of such intersection processes are not very clear.

9.5 Weak-fringing faults

Weak-fringing faults were observed in $<a>$ type slip bands. Faults observed were associated with slip bands on basal, prism and pyramidal planes. The magnitude of such faults were always weak on basal planes compared to faults both prism and pyramidal planes. The fault vector was identified to be of a “shear” type and always has its major component along the burgers vector. There is a small out-of-plane component to the fault vector. The direction of the fault vector was determined to be parallel to the burgers vector. In other words, the dislocations moving on the slip plane produce a displacement larger than the lattice translation along the $<a>$ direction. The magnitude of such a fault on the prism plane was quantitatively estimated using image matching simulations. These were estimated to be between $1/145[11\bar{2}0]$ to $1/104[11\bar{2}0]$, or about $0.06\AA$ to $0.085\AA$. These estimates are comparable to those that have been estimated in a Cu-15at% Al and in a $\gamma$-TiAl alloy. The weak-fringing faults are attributed to the creation incorrect nearest neighbors after the slip process. Hence, the origin of the fault is related to the SRO state. Presumably energy associated with this fault would be in addition to $\gamma_{\text{SRO}}$ discussed above.
9.6 Room temperature deformation anomalies

Several room temperature deformation anomalies have been observed in this study. Samples were observed to creep negatively when they were unloaded completely and allowed to recover any anelastic strain and then reloaded. No backflow was observed upon complete unloading, but negative creep was observed for a few tens of seconds after the reload. It is suggested that because the screw dislocations experience a large Peierls stress due their non-planar core structure, upon unloading they get locked, preventing any backflow. But, on subsequent reloading, in those regions which have large internal stresses the dislocations can backflow under load because the screw dislocations become mobile due to the applied stress. The duration of observation such negative creep is dependant upon the overall forward flow rate.

Large tension/compression asymmetry was observed in creep. There was a small difference in the 0.2% yield strength between tension and compression, with the sample being stronger in compression. But, sample crept in tension accumulated creep strains between 5 to 10 times greater than those observed in compression. This asymmetry was observed in both Ti-6Al and Ti-6242. Since the samples were well annealed before testing, a Bauschinger effect is ruled out. Further, it is not reasonable to expect similar internal stress states in both Ti-6Al and Ti-6242, which were processed very differently. Measurement of pole figures indicated that there is no strong texture in either alloy. It is suggested that the origin of this asymmetry might be related to generation and motion of \(<c+a>\) dislocations, because CRSS for \(<c+a>\) dislocations are much higher in compression than in tension. Since activation of \(<c+a>\) dislocations is important for slip accommodation across grains in a polycrystalline material, such a process could be easier
in tension than in compression. Another possibility is that the mobility of \(<a>\) type screw dislocations are higher in tension than in compression, although this explanation is not supported based on knowledge of the core structure of \(<a>\) type screw dislocations. As was discussed in chapter 8, the effect of aluminum addition on the \(<a>\) type screw dislocation core structure is not well understood. Therefore, one cannot rule out this second possibility.

Finally, room temperature recovery was observed in titanium alloys. Samples were deformed in creep and unloaded after some straining. They were kept at room temperature for a few months in the unloaded condition. When these samples were reloaded, the creep behavior was similar to a well-annealed sample. There was little evidence for the work-hardening that should have occurred during the prior creep. Room temperature recovery was observed in an AC sample as well as in a Ti-6242 sample. Such behavior was not observed when the samples were reloaded within 200h. Finally, when an IWQ sample was similarly tested and re-loaded, the creep behavior was a continuation of the prior creep response, with an initial transient period. Based on the deformation characteristics of these alloys it is suggested that the origin for this unusual behavior is due to the large stress concentrations at grain boundaries as result of the planar pile-ups. It is possible that with time some of these stress concentrations are relieved partly by activating dislocation sources in neighboring grains. When the sample is reloaded these new dislocations can become active and produce creep strains.
**10.1 Conclusions**

i) Using neutron diffraction, the presence of short-range order in a Ti-6Al alloy has been shown for the first time. The SRO state can be thought of as a precursor to the Ti$_2$Al ($\alpha_2$) phase.

ii) Phenomenological analysis of room temperature creep of titanium alloys indicates that this unusual behavior can be rationalized as due to a combination of abnormally low strain hardening exponent, $n$ and moderate strain rate sensitivity, $m$.

iii) The effect of SRO on room temperature creep was studied in a Ti-6Al alloy. These studies indicated that there are dramatic changes in the initial primary transients after various heat treatments to modify the SRO state. The long time behavior was similar among the different samples.
iv) Strengthening due to SRO was evaluated quantitatively in Ti-6Al. The energy of the diffuse APB was estimated to be between 17-48 mJ/m$^2$.

v) Weak fringing faults were observed in slip bands on basal, prism and pyramidal planes in Ti-6Al. These faults were always observed to be weak on the basal plane. The fault vector was observed to be of “shear” type, l/n<1120>. The dislocations causing the fault produce a displacement larger than a lattice translation along the <a> direction. It was also noted that the fault vector has a small component out of the slip plane in most cases and this is stronger in a few cases. The magnitude of the fault was estimated to be between 1/145<1120> to 1/104<1120>.

vi) Large asymmetry in creep response was observed between tension and compression in both Ti-6Al and Ti-6242. The alloys were observed to be more creep resistant in compression.

vii) Room temperature recovery was observed in both Ti-6Al and Ti-6242. Samples were crept to some strain and then left in the unloaded condition at room temperature for several months. When these samples were reloaded to the same stress level, they exhibited significantly increased creep rates (relative to those prior to unloading), indicating that the prior strain hardening had been partially recovered. The degree of recovery is dependent upon the duration in the unloaded condition.

10.2 Unresolved issues and Future work

Several fundamental questions regarding room temperature creep and deformation behavior of titanium alloys remain answered in this study. But several new phenomena
have been discovered and new questions have been raised which need to be resolved in order to develop a better understanding of the deformation behavior in these alloys.

a) One of the main arguments that has developed in this work to rationalize the observed creep transients is the dynamics of dislocation sources. It appears that differences in SRO are very subtle among the aged samples. It has further been argued that the source dynamics may in fact be very sensitive to frictional effects, as might result from slight changes to the SRO state. Hence, it will be very useful to perform dislocation dynamics simulation of such source operation. These simulations must include the different mobility of screw versus edge dislocations, as well as the effect of SRO. The comparison of these simulations with experimental results may provide further insight into the early stages of deformation.

b) Effect of SRO on room temperature creep was studied in detail. The SRO state after many of the heat treatments was determined using neutron diffraction. A direct correlation between the SRO state and the creep response has not clearly emerged for all the heat treatments. Detailed characterization of the SRO state by calculating Warren-Cowley SRO parameters for the different samples could provide more insight into the SRO state. Such studies could help in providing a better correlation between the creep response and the SRO state.

c) The diffuse APB created due to destruction of SRO, $\gamma_{SRO}$, was evaluated for prism slip bands. It was suggested that this quantity should be different on basal and pyramidal planes for the same SRO state. Such measurements need to be made to see whether this is
indeed true. Theoretical estimates of these quantities from measured SRO states should be made and compared to those estimated from dislocation structures.

d) It was postulated that one of the main reasons for the observed low work-hardening characteristics in titanium alloys is due to lack of significant work-hardening from slip bands intersections. Mechanistic details of such intersection processes are not clear at the present time. Such studies should help improve our understanding of the deformation characteristics of these alloys, and may provide additional directions to pursue for improving the properties of these alloys.

d) The weak-fringing fault due to supplementary displacements was analyzed in detail in chapter 7. It was shown that the magnitude of the shear fault is a small fraction of the atomic dimensions, and the nature of the fault is related to the SRO state. Further, the magnitude of the fault is always lower on the basal planes. An atomistic model explaining these observations needs to be developed. The energy associated with these faults also needs to be evaluated.

e) It was suggested that tension/compression asymmetry may be related to the mobility of $<a>$ type dislocations, and to the generation and motion of $<c+a>$ dislocations. It is suggested that the study of possible tension/compression asymmetry, and "non-schmid" effects in general, may be an extremely interesting topic for atomistic simulation. The role of $<c+a>$ dislocations in this effect, and their generation near grain boundaries, should be more thoroughly understood. Experiments exploring the tension/compression asymmetry as a function of grain size may be valuable for exploring more fully the role of $<c+a>$ dislocations.
f) The origin of the room temperature recovery phenomenon also needs to be understood better, by performing systematic studies of the time and strain-dependence of the recovery phenomenon. Studies of the internal stress as a function of static ageing time may also valuable as a means of characterizing the kinetics of the recovery processes which may be responsible for this effect.