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DATA ACQUISITION AND ANALYSIS OF TRANSIENT

CREEP DEFORMATION IN Ti-47A1-2Cr-2Nb

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Chih-Huei Wu

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Major professor Thomas R. Bieler

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DATA ACQUISITION AND ANALYSIS OF TRANSIENT CREEP DEFORMATION IN Ti-47Al-2Cr-2Nb

By

Chih-Huei Wu

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ABSTRACT

DATA ACQUISITION AND ANALYSIS OF TRANSIENT CREEP DEFORMATION IN Ti-47AI-2Cr-2Nb

By

Chih-Huei Wu

A low cost and high efficiency automated data acquisition was developed for transient creep testing. The data analysis was accomplished using a modified θ -projection equation for creep. The fitting parameters of the θ -projection equation exhibited similar stress exponent and thermal activation energy as steady state creep data.

Stress and temperature change creep experiments under a compression shear stress state were conducted on PST (Polysynthetically Twinned) and polycrystal (duplex) specimens of Ti-47Al-2Cr-2Nb at 25 ~87MPa and 760~810 ⁰C. Transient creep of PST and polycrystal after stress and temperature change exhibited 'normal' behavior and suggests Class M type creep deformation in TiAl. The stress exponent of n=5.6 and the activation energy of Q = 135 ± 70 KJ/mole for the PST indicating that dislocation glide aided by pipe diffusion is the dominant mechanism of creep deformation in this test regime. The values of n=2~3 and Q=229 ± 60 KJ/mole for polycrystal in Ar and n=2~3 and Q=574 ± 90 KJ/mole in air were obtained, respectively. Varying values of the stress exponent and the activation energy for the polycrystal may due to the effect of dynamic recrystallization, and a mixture mechanism suggesting that a composite mode is needed to describe the polycrystal creep behavior of TiAl.

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CHAPTER I

INTRODUCTION

Titanium Aluminide (TiAl) based materials are candidates for high temperature structural applications because of their low density, high specific strength, stiffness at elevated temperature and high resistance to oxidation and creep. For service at high temperature, it is very important to understand the creep behavior of TiAl based alloy. However, only limited research has been accomplished and there are still many mysteries regarding the mechanisms of creep in TiAl. The varied values of stress exponent (n=2~8) [1~13] and activation energy (80~600 KJ/mole) [1~13] suggest that complicated mechanisms of deformation are involved in the TiAl creep behavior. Therefore, some simple experiments which remove the effects of complicated microstructure are very helpful to understand the fundamental mechanisms of creep in TiAl.

The precise automated data acquisition system which consists of a personal computer with a data acquisition board provides a low cost, high efficiency and precise way to collect the data for creep test. The data analysis, combined with mathematical models that precisely characterize the creep curve, is very important in analyzing and interpreting the creep behavior.

The goals of current work are to build a precise data acquisition system for creep test and then, to conduct several stress and temperature change creep tests under compression shear stress on carefully oriented PST (Polysynthetically Twinned) and polycrystal specimens of γ -TiAl based alloy. The results of sophisticated stress and temperature

1

change creep tests will be analyzed using the power law equation and a modified θ -projection concept equation to examine the creep mechanisms from mechanical property experiments. These experiments will cover a range of stresses and temperatures so that stress exponents and activation energies can be obtained for these fundamental mechanisms.

These initial analysis is associated with the microscopy work which is in progress by colleagues. Deeper understanding of creep deformation mechanisms in TiAl will permit prediction of the creep life of TiAl in high temperature applications.

CHAPTER II

REVIEW OF CREEP

2.1 DEFINITION OF HIGH TEMPERATURE CREEP

Creep is defined as a time dependent deformation. Creep deformation mechanisms are strongly dependent upon temperature and applied stress. Different creep strain/time curves are usually found at different temperature (high or low). A typical low temperature (below about 0.4 T_m) creep strain/time curve is shown in Figure 2.1 [14]. Under low temperature conditions, the total creep strains are usually very low, typically much less than 1%, and the creep deformation rarely leads to failure. In general, creep is an important deformation phenomena at temperature conditions higher than half the melting temperature (T_m) in applications such as heat engines.

2.2 STAGES OF CREEP

In general, constant-load creep tests are used for engineering purposes. At high temperature the constant-load creep curve can be described by three stages: primary stage, secondary (steady state) stage, and tertiary stage (shown as Figure 2.2) [15]. In the primary stage, after the instantaneous strain ε_0 that is a sum of elastic and plastic deformation on loading, the creep strain rate decreases and then reaches an approximate constant and minimum value of creep rate during the secondary (steady state) stage. The steady state stage of creep is interesting from an analytical point of view because it represents most of the creep curve and most of the time of deformation, even though the creep strain may be



Figure 2.1 Strain/time curves for low temperature (T< 0.4T_m) [14]. (a) strain-time curve (b) strain rate-strain curve



Figure 2.2 Three stages of high temperature creep curve [15].

very small in the steady state stage. After reaching a constant strain rate for a period of time, the creep rate will increase again during the tertiary state which leads to fracture.

There are still many mysteries regarding creep properties, i.e. mechanisms of creep. Researchers [16,17] usually use dislocation phenomenon to describe the strain (work) hardening (generation of dislocation) and softening (recovery and annihilation of dislocation) in models of creep behavior. At high temperature ($T > 0.4T_m$), dynamic recovery starts to play a more important role than it does in low temperature. The dislocation density increase, i.e. work hardening, causes the creep to decrease with time in the primary stage. In the secondary stage, the creep rate reaches a steady state due to a balance between work hardening and dynamic recovery (work softening). The dynamic recovery can occur by various mechanism, i.e. dislocation glide, climb, cross slip,.....etc. The main mechanism is due to dislocation climb and annihilation (positive and negative signed dislocations cancel out each other). The work softening factor dominates in the tertiary stage and finally causes material fracture.

2.3 STRESS AND TEMPERATURE EFFECT

The shape of creep curve is strongly influenced by the temperature and stress. Figure 2.3 (a) [18] shows a series of creep curves corresponding to different stresses at the same temperature. In a similar manner, Figure 2.3 (b) shows that at constant stress, the shapes of creep curves change as temperature changes [18].

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(a)



Figure 2.3 (a) Stress effect and (b) temperature effect on the creep curve [18].

2.4 POWER LAW EQUATION

Considering both stress and temperature effects the empirical power law creep relation has been found to describe the steady state creep [14]

$$\dot{\varepsilon}_{s} = A\sigma^{n} \exp\left(\frac{-Q_{c}}{RT}\right) \tag{1}$$

where $\dot{\epsilon}_s$ is steady state strain rate and A is a constant, σ is the applied stress, Q_c is the activation energy for creep, R is the universal gas constant and T is the absolute temperature.

In some cases, there is no steady-state rate regime in the creep curve, and the minimum strain rate ε_m is usually used instead of $\dot{\varepsilon}_s$. The stress exponent value n can be obtained from the slope of $\log_e \dot{\varepsilon}_s$ vs. $\log_e \sigma$ plot at the same temperature. Similarly, a graph of $\log_e \dot{\varepsilon}_s$ vs. 1/T at constant stress will yield a straight line of gradient $-Q_c/R$ and activation energy can be calculated. The different mechanisms of creep in different stress/ temperature regimes will cause stress exponent n and activation energy Q_c values to vary.

By multiplying both sides of equation (1) by $exp(Q_c/RT)$, it can be rearranged as

$$\dot{\varepsilon}_{s} \exp\left(\frac{Q_{c}}{RT}\right) = A\sigma^{n} = Z \qquad (2)$$

The quantity Z is known as the Zener-Hollomon parameter [19] or as the temperaturecompensated creep rate [20,21]. By defining suitable value of Q_c , plots of log Z against log σ allow several creep curves for different temperatures to be superimposed onto a single line, as shown in Figure 2.4 [14]



Figure 2.4 The temperature-compensated secondary creep rate data (or Z) for polycrystalline copper. The activation energy of 130 KJ/mole is used for this figure. [14]

2.5 DEFORMATION MECHANISMS

In creep ($T > 0.4T_m$), depending upon the applied stress level (high, intermediate and low), there are two fundamental mechanisms of creep: diffusional creep and dislocation creep.

2.5.1 Diffusional creep

At low stress and high temperature, mass transport by diffusion of vacancies becomes the controlling mechanism and this creep is known as diffusional creep.

(a) <u>Nabarro-Herring creep</u>

Based on Nabarro-Herring theory of creep [22,23], creep occurs by stress-directed vacancy flow through the crystal lattice from grain boundaries in a state of local tension to grain boundaries with a local compressive stress, as shown in Figure 2.5(a) [24].

Nabarro-Herring creep can be used to describe the creep behavior which usually is found at low stress and high temperature. In general, the stress exponent n=1 and the activation energy $Q_c=Q_{SD}$ (self-diffusion) in those creep cases.

(b) <u>Coble creep</u>

In 1963, Coble [25] considered that the stress directed vacancy flow can go through grain boundaries instead of only through lattice as assumed by Nabarro and Herring. (see Figure 2.5(b)) [24]. In this case, the stress exponent n is also equal to 1 but the activation energy for creep is that for grain boundary diffusion, i.e., $Q_c = Q_{GB}$.



Figure 2.5 Schematic of (a) Nabarro-Herring creep (b) Coble creep[24].

2.5.2 Dislocation creep

When the temperature is between $0.4T_m$ and $0.7T_m$ and at the intermediate/high stress level ($10^{-4} < \sigma/G < 10^{-2}$), the dominant mechanism of creep is the rate of dislocation motion by glide or climb. This regime is known as dislocation creep. According to the dislocation rate-controlling mechanism, dislocation creep models can be divided into two types: dislocation glide and dislocation climb rate-controlled.

(a) <u>Dislocation glide rate-controlled</u>

The dislocation gliding in a crystal meets local obstacles, including precipitates and solute atoms; and if the impeding force of obstacles to dislocation glide is not strong, the dislocation may overcome the barrier by thermally-activated glide. Cottrell [26] and Weertman [27], who presented the models, assume that, in this case, dislocation glide is the creep rate-controlling process.

(b) <u>Dislocation climb rate-controlled</u>

The general idea of dislocation climb rate-controlled creep was developed by Weertman [28]. He considered dislocation climb as the recovery process. During deformation gliding dislocations which may come from different dislocation sources move across their glide planes until the leading dislocations are held up by obstacles on their slip planes, forming a pile-up array on the same slip plane. At high temperature the dislocations can pass those obstacles by climbing and then continue moving on a new glide plane. In such a glide-plus-climb sequence, the dislocation climbing is a slower process and therefore, it is a rate-controlling process (see Figure 2.6).



Figure 2.6 Schematic representation of the leading dislocation passes an obstacle by climbing [29]

2.5.3 Mechanisms map

Under different stress/temperature conditions, the dominant creep mechanism will change and cause the different creep behaviors. This type of information can be conveyed by construction of 'deformation mechanism map'[30] (see Figure 2.7).



Figure 2.7 Deformation mechanism map for a pure polycrystalline metal [30].

In general, theories to explain creep behavior can be based on diffusion creep and dislocation creep processes. We usually consider those two processes as independent process, and both of them contribute to the overall creep rate. Under different stress/temperature conditions, one of these processes contributes much more than that of the other. We define this process as 'dominant', and a different dominant mechanism leads to different stress exponent n and activation energy Q_c values, as shown in Table 2.1 [29].

Creep Process	Temperature	Stress	n value	Q _c value
high temperature dislocation creep	above ~0.7T _m	intermediate/high	> 3	~Q _{SD}
low temperature dislocation creep	~0.4 to ~0.7 T _m	intermediate/high	> 3	Q _{CORE}
high temperature diffusional creep (Nabarro-Her- ring)	above ~0.7T _m	low	~ 1	Q _{SD}
low temperature diffusional creep (Coble creep)	~0.4 to ~0.7T _m	low	~ 1	Q _{GB}

Table 2.1 Different n and Q_c values associated with dislocation and diffusional creep occurring with pure metals

 Q_{SD} : the activation energy for self diffusion

- Q_{CORE} : the activation energy for self diffusion along dislocation cores,
 - often termed pipe diffusion
- Q_{GB} : the activation energy for grain boundary diffusion

2.6 STRESS AND TEMPERATURE CHANGE CREEP

No matter what the rate-limiting dislocation creep mechanism is, most dislocation creep theories [16] state the secondary creep rate will vary with stress, temperature, grain size and dislocation structure. Stress or temperature change experiments result in transient creep behaviors which are used to provide some information about these microstructural factors. Temperature change has similar effect on the creep transient behavior of materials as stress change, but it is technologically more difficult to get good data, so in the following discussion, we only use the example of a stress change experiment to interpret those transient behaviors.

In general, the transient behavior after stress changes depends on the material and on the sign and magnitude of the stress change. In dislocation creep, there are two classified types: Metal (M) class in which dislocations can glide freely and Alloy (A) class in which is dislocations glide in a viscous manner. The different substructures and rate-controlling mechanisms cause those different creep behaviors.

2.6.1 Class M behavior

For Class M materials, the transient behavior is shown as Figure 2.8 (a) [32]. As stress increases, instantaneous strain is followed by transient deformation in which $d\epsilon/dt$ is much greater than the previous steady state rate, and decelerates to a new steady state. In this case, the instantaneous strain consists of an elastic and a plastic component. With stress decreases, an instantaneous elastic contraction is followed by transient creep, where $d\epsilon/dt$ is much less than the previous steady state rate and accelerates to a new steady state. This type is known as 'normal' transient behavior.



Figure 2.8 Schematic diagram of two types of creep transient after stress changes (a) Class M type (b) Class A type [32]. Δl_e : elastic strain, Δl_p : plastic strain

The Class M creep behavior is characterized by pronounced heterogeneous dislocation distribution during the normal primary creep. This is clearly shown by the subgrain structure formation. The subgrain structures initially form in the high stress concentration regions such as grain boundaries and then spread over the whole grain. The coarsening of small subgrains and refining of the coarse ones cause the heterogeneous dislocation density to decrease, and a very regularly spaced network of subgrain boundaries is developed.

Kuhlmann-Wilsdorf *et al.* [33] presented a theory that states that the equilibrium subgrain size depends on the dislocation density, and since the dislocation density is a function of applied stress, the relation between subgrain size and the applied stress can be found. Therefore, the transient phenomena resulting from changes of creep conditions such as stress changes or temperature changes are associated to the substructure changes. Lonsdale and Flewitt [34] showed that any change (reduction or increase) in applied stress results in a change in subgrain size that corresponds to the new applied stress, and this subgrain size is reached at the onset of the new steady state.

In general, the class M type creep is thought to be governed by the dislocation climb processes, i.e. dislocation glide contributes strain but the creep rate is controlled by the rate of dislocation climb processes in which n is generally close to 5. These glideclimb processes which are associated with the formation of substructure allow rearrangement and annihilation of dislocations to occur.

The dislocation movement during creep is also considered to be opposed by an "internal stress" or "dislocation back stress", denoted as σ_i . The internal stress σ_i generally depends on the dislocation structure, *i.e.* subgrain size, the applied stress, and testing temperature. The effective stress $\sigma^* = \sigma - \sigma_i$ is the difference between the applied stress σ

18

and the internal stress σ_i and describes the stress that causes dislocation movement.

The change in creep rate following a stepwise increase in the applied stress is thus due to an increase in the internal stress to a new equilibrium state, and consequently reducing the effective stress over time. Thus, after stress increases, the substructure rearrangement and annihilation of dislocations associated with decrease of effective stress σ^* cause deceleration of creep rate. On the other hand, after a stress reduction, the coarsening of the dislocation structure causes an increase of the effective stress for thermally activated glide. Thus, the strain rate increases after a stress reduction.

2.6.2 Class A behavior

For Class A materials, the transient behavior is shown in Figure 2.8(b) [32]. It is the 'inverse' of the normal transient behavior. In this case, the instantaneous strain is elastic.

In contrast to Class M materials, the subgrain structure is seldom or not observed in the Class A creep behavior. This means the dislocations remain homogeneously distributed with no formation of subgrain during the normal primary creep in the Class A materials.

In Class A materials, the dislocation glide becomes the slowest step due to the viscous drag exerted on a moving dislocation by the solute atmosphere, where n is equal to 3. Weertman [35,36] presented a glide-controlled model which assumes that the creep rate depends on the mobile dislocation density, ρ , as

$$\dot{\varepsilon} = \rho b \bar{v} \tag{3}$$

where \overline{v} is the average dislocation velocity that is determined by the rate at which the solute atoms can move along with the dislocations. **b** is the Burgers vector and ρ varies as the square of the applied stress. The solute-drag model can explain the inverse of normal primary creep behavior. A low dislocation density exists prior to creep, and the creep rate is limited by the mobile dislocation density; subsequent stress increases leads to an increased dislocation density, and therefore a higher creep rate. On the other hand, since no substructure forms and the solute atoms can move easily, they slow down the moving dislocation at lower stress, and the strain rate decelerates after stress decreases.

2.6.3 Incubation period and anelastic strain

There are two other phenomena which are usually observed. Figure 2.9 (a) shows "incubation periods" following large enough stress drops ($\sigma-\sigma_i < 0$, where σ_i is internal stress). It can be explained in term of the times taken for a dislocation network to readjust to a proper size for a new stress level [37]. Another phenomenon is time dependent, anelastic strain, following decreases in applied stress (see Figure 2.9(b)). Burton [37] describes this transient behavior as due to dislocations becoming unbowed after a sufficient stress drop, and give rise to a strain which is opposite in sense to the original strain.



Figure 2.9 Schematic diagram of (a) incubation period t_{inc} (b) anelastic strain ΔL_{an} [37]

CHAPTER III

ΤΗΕ θ–PROJECTION CONCEPT

In high temperature loading conditions, materials are subjected to long-term service under an applied stress and creep deformation occurs. In order to model this longterm creep behavior from short-term creep experiment data, people analyze creep deformation curves by some constitutive equations. The most famous equation is power-law equation which relates \dot{e}_s (steady state strain rate) to stress (σ) and temperature (T) by a power law relationship (see equation (1)). Unfortunately, the stress exponent (n) and activation energy (Q) are themselves functions of stress, strain and temperature so that different values of n and Q are observed as the test conditions are varied. By the way, the primary creep is an important issue for technological applications, and the steady state theory is not always helpful.

On the other hand, polynomial expressions have been used [38] and good fits can be obtained. However, it is difficult to interpret the polynomial coefficients in terms of physical meaning. In order to find a model which can not only accurately describe the shape of the creep curve but also relate to physical meaning of creep deformation, the θ projection concept has been proposed by Evans and Wilshire(1985) [39].

3.1 CONSTITUTIVE CREEP EQUATION

Based on the physical understanding of creep deformation in primary and second-
ary stages, the following constitutive equation is described as

$$\varepsilon = \varepsilon_i + \theta_1 \{ 1 - \exp(-\theta_2 t) \} + \dot{\varepsilon}_s t \tag{4}$$

where ε_i is the initial strain upon loading, and the second and third terms describe primary and secondary creep stages, respectively. $\dot{\varepsilon}_s$ is the secondary (or minimum) creep rate, and the parameters θ_1 and θ_2 are determined by curve fitting.

By differentiating equation (4) we can obtain

$$\dot{\varepsilon} = \theta_1 \theta_2 \exp\left(-\theta_2 t\right) + \dot{\varepsilon}_s \tag{5}$$

And the primary creep rate can be rearranged as

$$\dot{\varepsilon}_{p} = \theta_{1}\theta_{2}\exp\left(-\theta_{2}t\right) = \theta_{2}\left(\theta_{1} - \varepsilon_{p}\right)$$
(6)

where primary strain rate $\dot{\varepsilon}_p = (\dot{\varepsilon} - \dot{\varepsilon}_s)$. Equation (4) indicates a gradually decaying creep curve, and after a period of time a steady state stage ($\dot{\varepsilon}_s$) is reached. θ_1 is a parameter of strain-like meaning and θ_2 is a rate parameter as shown in Figure 3.1. From equation (5, 6), it shows that the primary creep rate ($\dot{\varepsilon}_p$) decreases gradually with increasing primary creep strain (ε_p) and approaches $\dot{\varepsilon}_s$ (steady state strain rate).

It is obvious that no tertiary creep is described by the above equation. All of the creep curve can be described by the following equation [39],



Figure 3.1 The θ projection concept envisages normal creep curves in terms of primary stage and secondary stage

.

$$\varepsilon = \varepsilon_i + \theta_1 \{ 1 - \exp(-\theta_2 t) \} + \theta_3 \{ \exp(\theta_4 t) - 1 \}$$
(7)

where ε_i is the initial strain upon loading, $\theta_1, \theta_2, \theta_3, \theta_4$ are determined by regression analysis of the whole creep curve up to rupture. θ_1 and θ_3 define the strain magnitude with respect to time, θ_2 and θ_4 describe the curvatures of primary and tertiary stages of creep, respectively. Evans and Wilshire [39] interpret the second term to represent the strain hardening term which dominates in primary creep, and the third term describes the strain weakening term which dominates in tertiary creep. The secondary stage is a consequence of dynamic balance between strain hardening (second term of equation (7)) and the strain weakening term (third term of equation(7)). It agrees qualitatively with theoretical interpretation of creep deformation by dislocation motion. However, a true steady state creep in the secondary stage is not predicted by this model; secondary creep is only a transition between primary and tertiary creep processes.

3.2 MODIFICATIONS OF 0-PROJECTION CONCEPT

Maruyama *et al.* [40] thought the initial strain ε_i is the least reliable value of measurement even when high-precision test methods are used. It is because ε_i is determined by calculating only the difference in strain immediately before loading and immediately after loading. Therefore, in order to get a better interpretation, the following equation is

presented by Maruyama et al. [40],

$$\varepsilon = \varepsilon_o + A \{ 1 - \exp(-\alpha t) \} + B \{ \exp(\beta t) - 1 \}$$
(8)

where ε_0 is an adjustable parameter instead of initial strain in the original θ -projection model. A, a, B and b are the parameters of curve fitting. α represents the rate constant of strain hardening process and β represents the rate constant of strain weakening process.

Maruyama *et al.* [41] made further modifications in 1987. They discarded those creep data after a certain cutoff strain $\varepsilon_{a=b}$ when determining the parameters of equation (8) and simplified to,

$$\varepsilon = \varepsilon_0 + A \{ 1 - \exp(-\alpha t) \} + B \{ \exp(\alpha t) - 1 \}$$
(9)

which has only one rate constant α instead of the two rate constants in equation (8). The same rate constant α for both strain hardening and weakening processes is not suitable for all materials due to complicated mechanisms during creep deformation. Actually, two rate constants may be more proper than one rate constant since changes in the microstructure lead to changes in the rate-controlling mechanism during the creep process.

3.3 INTERPRETATION OF THE PARAMETERS

In order to interpret those parameters obtained from curve fitting, the linear stress/ log θ_1 and temperature/log θ_1 plots have been used.

3.3.1 Hardening and weakening parameters: θ_1 and θ_3 (or A and B)

Brown *et al.* [39] found θ_1 and θ_3 in equation (7), which govern strains associated with the primary and tertiary stages respectively, can be rationalized simply by normalizing the creep stress with respect to the Young's modulus (or yield stress) at the creep temperature. (see Figure 3.2 (a)) [42]. Thus, the strain-related terms θ_1 and θ_3 are temperature and stress dependent. They found a similar behavior pattern in: 1/2Cr1/2Mo1/2V steel [39], type 316 austenitic steel [43] and cast superalloy IN 100 [44]. On the other hand, Maruyama *et al.* [45] also state that weakening parameter B in equation (8) is a function of

$$f_b(\sigma/E)exp(-Q_b/RT)$$

where $f_b(\sigma/E)$ is a function only of σ/E , $Q_b=2$ ($Q_c - Q_d$) and Q_c is activation energy for minimum creep rate, Q_d is activation energy for self-diffusion. But for CrMoV steel, they found that the hardening parameter A is invariant with temperature and only a function of (σ/E).

3.3.2 Rate constant parameters: θ_2 and θ_4 (or α)

Both Brown *et al.* [39,44] and Maruyama *et al.*[43,45] concluded these rate constants can be expressed as (see Figure 3.2(b))

$$\theta_2, \theta_4 \text{ or } \alpha = f_{\alpha}(\sigma/E) \exp(-Q/RT)$$
 (10)

where σ is the creep stress, E is Young's modulus, $f_{\alpha}(\sigma/E)$ is a function independent of temperature and Q is the activation energy for the rate constant. It is expected that the rate constants θ_2 , θ_4 or α are closely related to the rate-controlled step of creep. In other words, the activation energy for those rate constants are associated with the dominant mechanism



(b)

Figure 3.2 Rationalization of (a) θ_1 and θ_3 (b) θ_2 and θ_4 for polycrystalline copper [42]

of primary or tertiary stage of creep. However, further understanding of the relationships between rate constant and the rate limiting deformation mechanism still needs more work.

3.3.3 Minimum strain rate

By differentiating equation (7), the strain (creep) rate at any condition is

$$\frac{d\varepsilon}{dt} = \theta_1 \theta_2 \exp\left(-\theta_2 t\right) + \theta_3 \theta_4 \exp\left(\theta_4 t\right)$$
(11)

There is a minimum gradient (strain rate) at a time t_m which is the time to reach the minimum strain rate, expressed as

$$t_m = \frac{1}{\theta_2 + \theta_4} \ln \left(\frac{\theta_1 \theta_2^2}{\theta_3 \theta_4^2} \right)$$
(12)

Thus, if the θ_i terms are known for a set of testing conditions, the t_m can be calculated by equation (12) and therefore the minimum creep rate ε_m can be obtained.

3.3.4 Prediction of rupture life

The four θ functions vary with stress and temperature and can be defined by using the following equation: [44]

$$\log_{10} \theta_{i} = a_{i} + b_{i}T + c_{i}\sigma + d_{i}\sigma T \quad (i=1,2,3,4)$$
(13)

where a_i, b_i, c_i, d_i are constants. Similarly, by this approach, the fracture strain ε_f can be expressed as

$$\varepsilon_{\rm f} = a + bT + c\sigma + d\sigma T \tag{14}$$

where a,b,c,d are constants. Those coefficients describing ε_f (a,b,c,d)and four θ functions (a_i,b_i,c_i,d_i) can be computed by multilinear least squares regression analysis. The relevant rupture life is defined as the time t_f taken to reach the ε_f (fracture strain) under a particular stress and temperature condition. Therefore, ε_f can be computed by equation (14) and t_f can be obtained by solving the following equation:

$$\theta_1 \{ 1 - \exp\left(-\theta_2 t_f\right) \} + \theta_3 \{ \exp\left(\theta_4 t_f\right) - 1 \} - \varepsilon_f = 0$$
(15)

3.4 MODIFICATION FOR OUR ANALYSIS

For our experiments, we are interested in the primary and secondary stage of TiAl creep deformation, but especially the primary stage creep. The tertiary and rupture life are beyond our goals and therefore, we choose equation (4) as the constitutive equation for analyzing those curves of primary (or secondary) creep. However, a modification of equation (4) due to different starting time for a particular different test condition following a stress or temperature increase was used. The modified equation for transient creep after stress and temperature increase is

$$\varepsilon = \varepsilon_0 + A \left\{ 1 - \exp\left(-\alpha \left(t - t_0\right)\right) \right\} + \dot{\varepsilon}_s \left(t - t_0\right)$$
(16)

where ε_0 is an adjustable parameter, t_0 is starting time. $\dot{\varepsilon}_s$ is the steady state (or minimum) strain rate, A and α are curve fitting parameters. In the case of transient creep after stress

and temperature decrease, the constitutive equation is

$$\varepsilon = \varepsilon_0 + A \exp\left(-\alpha \left(t - t_0\right)\right) + \dot{\varepsilon}_s \left(t - t_0\right)$$
(17)

By differentiating equation(16) it can be rearranged as

$$\dot{\varepsilon} = A\alpha \exp\left(-\alpha\left(t - t_0\right)\right) + \dot{\varepsilon}_s \tag{18}$$

The equation (18) implies that the creep rate during the primary stage decreases gradually towards a definite "steady state" value, $\dot{\mathbf{e}}_{s}$, in the period of time t-t₀. However, these modified equations (16,17) are not suitable to fit the transient creep behavior during the temperature and stress change, as they are applicable only for constant stress and temperature conditions.

CHAPTER IV

REVIEW OF TIAL CREEP

TiAl is being considered as a candidate material for high temperature gas turbine jet engines due to its superior strength-to-weight ratio and good high temperature strength. Recently researchers have become more interested in the TiAl/Ti₃Al two-phase compounds rather than the single-phase Al-rich TiAl compounds because of the better ductility of the two-phase compounds. The compounds of TiAl with slightly Ti-rich compositions exhibit a two-phase microstructure which consists of the TiAl γ phase and a small volume fraction of the Ti₃Al α_2 phase. These are often called near-gamma alloys.

4.1 STRUCTURE OF TIAI

4.1.1 Crystal lattice structure

There are two basic lattice structures usually found in TiAl. One is γ phase (TiAl) with L1₀ (FCT) structure (see Figure 4.1(a)) [46], and the other is α_2 phase (Ti₃Al) with DO₁₉ (HCP) structure (see Figure 4.1(b)) [46].

The γ -TiAl has L1₀ type lattice crystal structure where the titanium and aluminum atoms alternately stack in sequence of ABCABC on (002) planes. It is a face-centered tetragonal structure with c/a ratio=1.02 that changes as Al content changes. The anisotropic crystal structure causes more complicated slip in the L1₀ lattice [47].

The α_2 (Ti₃Al) has DO₁₉ crystal lattice structure where the stacking sequence is



Figure 4.1(a) $L1_0$ lattice structure [46]



Figure 4.1(b) DO₁₉ lattice structure [46]

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ABABAB. The DO_{19} structure is based on the hexagonal closed-packed structure (HCP), but with a larger value of basal plane axis (2a) (see Figure 4.1 (b)) [46]. However, the system is often off-stoichiometric with Al atoms in Ti sites and therefore, this causes a slightly disordered lattice.

The lamellar structure occurs in the two-phase TiAl alloys with nearly stoichiometric or Ti-rich composition. The lamellar structure is composed of alternating laths of gamma(γ)-TiAl phase and α_2 -Ti₃Al phase with the orientation relationship of [48]

$$(111)_{\text{TiAl}} \parallel (0001)_{\text{Ti}^3\text{Al}}, <110 >_{\text{TiAl}} \parallel <11\overline{2}0 >_{\text{Ti}^3\text{Al}}$$

The lamellar boundaries are parallel to (111) in TiAl (γ) and (0001) in Ti₃Al(α_2). OH *et al.*[49] concluded the γ -phase has six possible orientations of [110] on (111) with respect to <11 $\overline{2}$ 0> on (0001) in the α_2 phase.

4.1.2 Microstructure

Since single phase γ -TiAl suffers from low ductility and toughness at ambient temperature, many efforts have been made in recent years to develop two-phase γ/α_2 alloys based on Ti-(49~51)at%Al [50], due to the higher ductility and toughness displayed by this stoichiometry.

Depending upon the heat treatments, there are four types of microstructures in these alloys: (a) fully lamellar (b) nearly lamellar (c) duplex (d) nearly-gamma. The different heat treatment conditions and features of the four types of microstructures are summarized in Table 4.1 [51] and illustrated in the corresponding phase diagram of the central region is shown in Figure 4.2 [52].

Microstructure	Heat-treatment condition	Features
Fully lamellar	in the single phase α field	 *large grain size ~> 500 µm with alternating γ and α₂ platelets *most brittle at room temperature, but excellent creep resistance
Nearly lamellar	just below T_{α} in the $\alpha + \gamma$ region	*coarse lamellar grains and a small vol- ume fraction of fine γ grains
Duplex	equal amounts of α and γ in α + γ region	*grain size ~15 μ m which consists both single phase γ equi- axed and lamellar (γ + α_2) structure *has the highest duc- tility at room tempera- ture
Nearly-γ or γ	low temperature in the $\alpha + \gamma$ region	*grain size ~13-50 μ m and banded regions consist of fine γ and α_2 grains

Table 4.1 The different heat treatment conditions and features for four types microstructures in TiAl based alloy [51]



Figure 4.2 The central part of the Ti-Al equilibrium phase diagram [52].

The creep properties of TiAl-base alloys are strong sensitive to the microstructure [53]. Huang [54] found the fully lamellar microstructure has most excellent creep resistance but poor ductility. On the other hand, the duplex microstructure is superior in ductility but poor in fracture and creep resistance. The details of creep mechanisms in different microstructure regions still need more investigations.

4.1.3 PST crystal

The two-phase TiAl with nearly stoichiometric or Ti-rich composition were usually composed of randomly oriented grains which have lamellar structure (γ + α_2). However, if this material is remelted and resolidified in a specific direction and an appropriate rate, the single-crystal like crystals which consist of only a single α grain orientation with lamellar structure can be obtained. Fujiwara *et al.* [55] identify this material with singlecrystal like structure as Polysynthetically Twinned (PST) crystal, since it contains a lot of thin twins that are all parallel to the lamellar boundaries.

Since complicated effects of microstructure variation exist in near γ -TiAl alloys, OH *et al.* [49] investigated deformation behavior in PST crystals which have the same microstructural features of lamellar grains, and obtained fundamental information about near- γ TiAl deformation. They found there are two modes of deformation in PST crystal. One is the shear deformations across the lamellar boundaries (hard mode) and the other is the shear deformations parallel to the lamellar boundaries (easy mode). By systematic studies of PST crystal deformation, the lamellar orientation dependence of deformation behaviors were found. Furthermore, a PST crystal with a specific orientation of lamellar boundary can provide useful information of how the lamellar structure affects the creep deformation in the near- γ TiAl alloys.

4.2 ALLOYING

The general composition of γ -TiAl which has been studied is titanium, 46~52 at% aluminum, and 1~10 at% M, where M represents at least one element from V, Cr, Mn, W, Mo, Nb and Ta [50]. Huang and Hall [56] found the highest ductility at room temperature of γ -TiAl is in binary TiAl alloys which have Al concentrations of 46~51 at% with the peak occurring at about 48 at% Al. The main purpose of adding alloys is to improve ductility (*e.g.* V, Cr) and oxidation resistance (*e.g.* Nb, Ta). These effects of adding alloys are listed in Table 4.2 [57].

4.3 CREEP IN TIAI

The mechanisms of creep deformation in TiAl are very complicated and so far only limited knowledge has been obtained. Based on those results, creep behavior in TiAl will be affected by the composition, microstructure, grain size and test conditions (stress and temperature). The microstructural variables include the structure of the lamellar grain boundaries, spacing of the α_2 - γ laths, and the nature of the α_2 - γ lath interface. However, there are still some problems of understanding creep behavior in TiAl: (1) no steady state creep regime is observed; (2) no instantaneous strain or primary creep is seen for some composition; (3) dynamic recrystallization occurs at low strains.

Elements	Properties are effected by this element
Al	It strongly affects ductility by changing the microstructure. Best ductil- ity occurs in the range of 46-50 at%
В	Add >0.5 at% refines grain size, and improve strength and workability.
С	Carbon-doping increases creep resistance and reduces ductility
Cr	Add 1-3 at% increases the ductility of duplex alloys Add >2 at% enhances the workability and superplasticity Add >8 at% greatly improves the oxidation resistance
Er	Changes the deformation substructures and increase the ductility of single-phase gamma
Fe	Increases fluidity, but also the susceptibility to hot cracking
Mn	Add 1-3 at% increases the ductility of duplex alloys
Мо	Improves the ductility and strength of a fine-grained material Improves the oxidation resistance
Ni	Increases fluidity
Nb	Improves greatly the oxidation resistance and creep resistance slightly
Р	Improves oxidation resistance
Si	Add 0.5-1 at% improves the creep and oxidation resistance Improves fluidity, but reduces the susceptibility to hot cracking
Та	Improves the creep and oxidation resistance Increases the susceptibility to hot cracking
v	Add 1-3 at% increases the ductility of duplex alloys and reduces the oxidation resistance
W	Greatly improves the oxidation resistance Improves the creep resistance

Table 4.2 Alloying effects the properties of TiAl [57]

4.3.1 No steady state regime

Most researchers [6,58,59] found no steady-state strain rate regime in TiAl creep behavior. Most observations of creep of near- γ TiAl-based alloys reached a minimum strain rate followed by an increasing strain rate. Thus, the minimum strain rate is usually used to analyze the creep behavior in TiAl.

4.3.2. No instantaneous strain

Based on the result of Oikawa's *et al.* [3~5], there is no instantaneous strain observed in equiaxed gamma (Ti-53Al). A limited strain in the primary stage is observed, followed by an apparently steady state strain region. This type of creep behavior is unusual, and the mechanisms of this creep behavior have not been explained yet.

4.3.3 Dynamic recrystallization

Dynamic recrystallization (DRX) has been reported [8] to be an important phenomenon at higher temperature (~760 °C) and at strains >~5% strain. The dynamic recrystallization nucleates along prior grain boundaries of the original microstructures and forms very fine grains at grain boundaries [58]. The dynamic recrystallization causes no steady state creep rate to be observed, and a minimum creep rate was obtained following a steep increase in the creep rate of most near- γ TiAl based alloys. The minimum creep rate is affected by the easiness of dynamic recrystallization, and this may be one reason why the varied values of activation energy (80~600 KJ/mole) in TiAl were obtained [3~8,54~57]. The activation energy of the characteristic steady state associated with dynamic recovery (269 KJ/mole) is usually lower than the activation energy of the apparent steady state during DRX (400 KJ/mole) [8].

4.4 MECHANISMS OF CREEP IN TIAI

There are two fundamental mechanisms of creep in TiAl: dislocation controlled creep or diffusion controlled creep. The dominant mechanism depends on the temperature and stress condition. Both of these mechanisms have been found in the creep behavior of TiAl. [1~13].

4.4.1 Diffusion controlled creep

In many cases, the activation energy for creep is associated with the activation energy of self-diffusion (lattice diffusion). The activation energy of ~291 KJ/mole was found by Kroll, Mehrer [60] for the self-diffusion in Ti-54Al. However, Ti-54Al has a near- γ TiAl structure which is composed mostly of gamma phase without much α_2 phase. Thus, the activation energy value of lamellar-based TiAl may be different from that of near- γ structure. Actually, a fundamental description of self diffusion in gamma TiAl with the activation energy has not been determined yet. The generally accepted value for the activation energy for grain boundary diffusion is 0.6 times the value for self-diffusion, i.e. $Q_b=0.6Q_L$, where Q_b is the boundary diffusion activation energy and Q_L is the lattice diffusion activation energy, respectively. The Q_b is ~202 KJ/mole and Q_L is ~336 KJ/mole for gamma TiAl were calculated by Ashby [61].

However, the measured activation energies for creep deformation on single phase alloys (423~560 KJ/mole) [62,63] are usually higher than that required for interdiffusion within single phase gamma (80~168 KJ/mole) [9,64]. This suggests that creep behavior in

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 γ -TiAl based alloys can not just be explained by intrinsic diffusional processes only. Phenomena such as dislocation/cell structure, pipe diffusion and dynamic recrystallization significantly affects the creep behavior of γ - TiAl based alloys.

4.4.2 Dislocation controlled creep

At higher stress conditions, dislocation creep is the dominant deformation mechanism in TiAl creep [7,8,59]. Twinning deformation is also an important deformation mechanism during creep of TiAl [47].

(a) Dislocation climb

Hayes and McQuay [12] investigated a γ -TiAl alloy with a fully transformed structure deformed in the temperature range 769~861°C and stress range 69~310 MPa. The creep behavior of the γ -TiAl alloy was a dislocation climb-controlled process which was aided by diffusion. Stress exponent n of ~5 and activation energy of ~335 KJ/mole was found. Hayes and London [7] also reported in the temperature range 704 ~850 °C and stress range 103.4~241.3 MPa for Ti-48Al-1Nb γ -TiAl with duplex microstructure, the activation energy is 326.4 KJ/mole and a stress exponent n=4.95. The value of the stress exponent is in agreement with dislocation climb process and the value of activation energy is also within the range for γ -TiAl alloys where creep rate is controlled by volume diffusion. Thus, it suggested that if dislocation climb is the rate-controlled mechanism, the expected creep activation energy is close to that of self diffusion and a stress exponent is in the range of 4~5. In general, the dislocations in TiAl creep specimens are 1/2<110] normal dislocations [55].

(b) Twinning

Mechanical twinning during creep deformation in TiAl has been reported by several researchers [3,58,65,66]. Twinning is known as the dominant deformation mechanism in TiAl for many crystal orientations. During primary creep, the fine mechanical twins initiate from a grain boundary at the intersections between horizontal twin layers and the grain boundary and grow into the grain interior. The continuous emission of twinning dislocations which were identified to be $1/6 < 11\overline{2}$] Shockley partials were found to be emitted from the grain boundary due to the local stress concentration. If the local stress were large enough to keep moving those dislocations, the propagation of the fine mechanical twins across the entire grain to reach the opposite grain boundary will occur. Fine mechanical twins were formed at the grain triple points during creep [66].

The contribution of mechanical twinning to creep deformation in TiAl is significant at a relatively low temperature and a high stress. In general, mechanical twinning during creep is significantly increased as the creep temperature decreases. Huang and Kim [11] found no evidence of mechanical twinning during creep deformation at 900 °C. On the other hand, mechanical twinning has more important effects on the creep deformation in TiAl at a higher stress.

Jin [67] also found 1/2 < 110] normal dislocation in TiAl creep. According to his researches in Ti-48Al-2Nb-2Cr (duplex), subboundaries were observed both in equiaxed gamma grains and within gamma laths in lamellar grains after a multi-stress drop test at 765 °C. By those analyses of subboundaries, the creep deformation mode in TiAl was identified as pure metal type (Class M).

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4.4.3 Composite model

Since the mechanisms of creep of TiAl are complicated, the models of composite theory were used to help interpretation. The composite model is based on the contributions of different individual mechanisms and determined by using a simple rule-of-mixtures approach. For the fully lamellar Ti-48Al-2Cr-2Nb at the high temperature, Hofman and Blum [31] presented a simple composite model which assumes that the total strain rate results from the local strain rates \dot{e}_{g} in the globular and \dot{e}_{1} in the lamellar regions as

$$\dot{\varepsilon} = (1 - f_l) \dot{\varepsilon}_g + f_l \dot{\varepsilon}_l \tag{19}$$

where f_1 is the volume fraction of lamellar regions and $\dot{\epsilon}_g$ and $\dot{\epsilon}_l$ are expressed by the density ρ_f and the velocity γ of free dislocation:

$$\dot{\varepsilon}_{j} = \frac{b}{M} \rho_{j} B(T) (\sigma_{j}^{*})^{m} \qquad j=g, 1 \qquad (20)$$

where M=3 is the Taylor factor, **b** is burgers vector, B(T) and m are constants. The effective stresses σ^* are determined by the difference between applied stress and internal stress and can be expressed as:

$$\sigma_{g}^{*} = \sigma - \alpha M G b \sqrt{\rho_{f}}$$
⁽²¹⁾

$$\sigma^*{}_l = \sigma - \alpha MGb \sqrt{\rho_f} - \sigma_l \tag{22}$$

where α is constant, G is shear modulus and σ_l represents the amount of the strengthening contribution of the lamellae.

Another composite model was presented by Soboyejo and Lederich [10]. In their model, the creep behavior of gamma based alloys with duplex α_2 and γ structure can modelled by recognizing that the two-phase alloys are composites that consist of α_2 and γ phases. By the composite theory and rule-of-mixtures, the total strain rate is determined by the contributions from alpha2-gamma phase and gamma phase in which a number of mechanisms contribute to creep deformation. The composite strain rate is thus given by

$$\dot{\varepsilon}_{c} = f_{\alpha_{2}} \left[\dot{\varepsilon}_{DC_{\alpha2}} + \dot{\varepsilon}_{DG_{\alpha2}} + \dot{\varepsilon}_{C_{\alpha2}} + \dot{\varepsilon}_{NH_{\alpha2}} \right] + f_{\gamma} \left[\dot{\varepsilon}_{DC_{\gamma}} + \dot{\varepsilon}_{DG_{\gamma}} + \dot{\varepsilon}_{C_{\gamma}} + \dot{\varepsilon}_{NH_{\gamma}} \right]$$
(23)

where subscripts DC, DG, C, NH represent the dislocation climb, dislocation glide, Coble creep, and Nabarro-Herring creep, respectively, and f is the phase volume fraction.

4.5. SUMMARY OF CREEP DATA FOR SEVERAL γ -TIAI ALLOYS

Only limited knowledge on the creep behavior exists for γ -TiAl since it varies with composition, stress, temperature and microstructure. Table 4.3 shows power law creep constants for several γ -TiAl alloys.

Alloy(at.%)/ processing	Microstructure	σ range (MPa)	Temp (⁰ C)	n	Q (KJ/ mole)	REF
Ti-50 Al/HIM/ As-cast	Lamellar+y at colony bound- aries	100-251	677-877	7.9	359	1
Ti-50.3 Al/PM/ HE at 1413 ⁰ C +HT	Lamellar + γ d _{γ} = 60 μ m; col- ony:50-100 μ m	103-241	700-950	4	300	2
Ti-53.4 Al	Equiaxed γ	80-400	677-927	8.0(L) 4.5(H)	600 360	3~5
Ti-50Al/com- pression creep	Equiaxed γ, d=179-90 μm	100-400	827	7.7	600	6
Ti-48.7 Al-2.2 W/same as Ti- 50.3 Al/PM	Lamellar; γ at colony bound- aries; W-rich par- ticles at phase boundaries	103-241	700-950	5.5	370	2
Ti-48 Al- 1Nb(0.09 wt% O)/ISR+HIP	Duplex	103-241	704-850	5-4.46	320- 341	7

Table 4.3 The creep properties of several γ -TiAl alloys

Alloy(at.%)/ processing	Microstructure	σ range (MPa)	Temp (⁰ C)	n	Q (KJ/ mole)	REF
Ti-48Al-2Cr- 2Nb/VAR+HIP	Duplex	103-300	705-815	3(L) 7(H)	300(L) 410(H)	8
Ti-48Al-2Cr/ HIM	Duplex	330	800	4.3	460	9
Ti-48Al-2Cr/ HIM	Nearly lamellar	200	700 900	2.4 3.5	292 292	10
Ti-47Al-1Cr- 1V-2.5 Nb/ Cast+HIP+extr usion at 1290 ⁰ C. HT:1360 ⁰ C+1000 ⁰ C	Fully lamellar, fine γ at colony boundaries	138-276	900	5.8	ND	11
Ti-48Al-2Mn- 2Cr	Fully lamellar	69.4- 310	760-871	4.6	335	12
Ti-49.5Al- 2.5Nb-1.1Mn/ cast +HIP	Equiaxed γ, d=125 μm	75-200	850-1377	ND	411	13

Table 4.3 The creep properties of several γ -TiAl alloys (cont'd)

CHAPTER V

DATA ACQUISITION SYSTEM

The automated data acquisition system is used for creep testing based on two reasons: time-savings and precise data are needed. Thus, an automated data acquisition system, consisting of a PC with data acquisition board, was developed for our creep experiments.

5.1 HARDWARE

The hardware of our automated data acquisition system consists of a computer and data acquisition board (DAB).

5.1.1 Computer

An IBM PS/2 model 30 with 80286 microprocessor is used. The benefits of PCcompatible machines are their low cost, expandability and relatively fast processor speed. Computer performance was enhanced by an Intel 80287 math coprocessor and 4 Mbyte of expansion RAM. Peripheral devices included 20 Mbyte hard disk and an IBM dot matrix printer. A sketch of the system is shown in Figure 5.1.

5.1.2 Data Acquisition Board

The Data Acquisition Board (DAB) used is the AT-MIO-16 made by National Instruments Corporation. The AT-MIO-16 board is a high-performance multifunction



Figure 5.1 The sketch of creep test units with data acquisition system

analog, digital and timing I/O board for PC. It contains a 12-bit ADC (Analog-Digital Converter) with up to 16 analog input channels, two 12-bit DACs (Digital-Analog Converter) with voltage outputs, eight lines of TTL-compatible digital I/O, and three 16-bit counter/timer channels for timing I/O. The basic data acquisition system operation is shown in Figure 5.2 [69].

One of the goals of current work is to build a precise data acquisition system for creep test. Therefore, in the following pages, those important issues related to the precision of creep data, data acquisition time, and noise will be mentioned and discussed.

(a) Analog input

The AT-MIO-16 board has three different input modes: non-referenced singleended (NRSE) input, referenced single-ended (RSE) input, and differential (DIFF) input. The single-ended input configurations use 16 channels with common analog ground. The DIFF input configurations use 8 channels where each input signal has its own reference and the difference between each signal and its reference is measured. On the other hand, for NRSE input mode, which uses 16 channels, all the input signals are referenced to the same common mode voltage, but this common mode voltage is allowed to float with respect to the analog ground of the board. For the RSE input mode, it also uses 16 signal input channels as the NRSE mode does, but the RSE mode refers all input channels to a common ground point that is tied to the analog input group of the AT-MIO-16 board rather than floating with respect to ground of the board (NRSE mode).

The AT-MIO-16 board offers two input range conditions: unipolar and bipolar input and two input ranges: 10V and 20V. The actual input range depends on the polarity, range configuration and gain (see Table 5.1) [70]. The precision values in Table 5.1

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Figure 5.2 Basic data acquisition system block diagram [69]

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Range Configuration	Gain	Actual Input Range	Precision*
0 to +10 V	1	0 to +10 V	2.44 mV
	2	0 to +5 V	1.22 mV
	4	0 to +2.5 V	610 μV
	8	0 to +1.25 V	305 μV
	10	0 to +1 V	244 μV
	100	0 to +0.1 V	24.4 μV
	500	0 mV to +20 mV	4.88 μV
-5 to +5 V	1	-5 to +5 V	2.44 mV
	2	-2.5 to +2.5 V	1.22 mV
	4	-1.25 to +1.25 V	610 μV
	8	-0.625 to +0.625 V	305 μV
	10	-0.5 to +0.5 V	244 μV
	100	-50 mV to +50 mV	24.4 μV
	500	-10 mV to +10 mV	4.88 μV
-10 to +10 V	1	-10 to +10 V	4.88 mV
	2	-5 to +5 V	2.44 mV
	4	-2.5 to +2.5 V	1.22 mV
	8	-1.25 to +1.25 V	610 μV
	10	-1 to +1 V	488 μV
	100	-0.1 to +0.1 V	48.8 μV
	500	-20 mV to +20 mV	9.76 μV

Table 5.1 Actual range and measurement precision versus input range selection and gain [70]

* The value of 1 least significant bit (1 LSB) of the 12-bit ADC, that is, the voltage increment corresponding to a change of 1 count in the ADC 12-bit count.

represents the value of 1 least significant bit (LSB) of the 12-bit ADC, the voltage increment corresponds to a change of 1 count in the ADC 12-bit count.

(b) Precision

For a 12-bit A/D converter, the 12-bit resolution allows the converter to resolve the input range into 4096 different steps (2^{12} =4096). For example, the least significant bit (LSB) for 20V, bipolar input range with gain = 1 is

$$LSB = 20 / 2^{12} = 4.88 \text{ mV}$$

The precision of this input range and gain combination is 4.88 mv. The value of LSB (precision) vary with the value of input range and gain. As the Table 5.1 shows, the higher gain value, the higher resolution (precision) can be obtained.

(c) Data acquisition

The data acquisition is controlled by the onboard sample counter. There are three data acquisition possibilities: single-channel data acquisition, multiple-channel (scanned) data acquisition with continuous scanning and multiple-channel data acquisition with interval scanning. For single-channel data acquisition, all A/D conversion data are read from a single channel. The multiple-channel data acquisition performs a multiple-channel scanning which is controlled by the multiplexer (mux) counter and the mux-gain memory. The continuous scanning means scanning cycles through the mux-gain memory without any delays between cycles. On the other hand, interval scanning assigns a time interval which is called the scan interval to each cycle through the mux-gain memory.

Data acquisition rates (number of samples per second) are determined by the conversion period of the ADC plus the sample-and-hold acquisition time. The rate varies from 37,000 samples/sec to 100,000 samples/sec with scanning mode (single or multiple channel) and type of board (high level or low level).

The data acquisition rates (sampling rate) should be a proper rate which can reconstruct the sampled signal accurately without collecting unnecessary information. According to the Shannon and Nyquist sampling theorem [71], the sampling rate must be greater than twice the highest frequency component of the waveform being sampled to allow the signal being sampled to be reconstructed accurately.

5.2. SOFTWARE

The AT-MIO-16 board can be controlled by Labwindows software provided by the National Instruments Corporation. The Labwindows enhances Microsoft QuickBASIC and C with an interactive development environment, function panels to generate source code, and a library for data acquisition, instrument control, data analysis and presentation. By programming QuickBASIC with Labwindows libraries, a window user interface application of data acquisition for creep testing was developed (see Figure 5.3).

5.3 NOISE

Since accurate data are needed for a creep experiment, especially for stress and temperature change processes (need record 1 point /per mV changes), the error caused by noise has a significant effect on the recorded data.

Unfortunately, signals are affected by noise in the laboratory environment, even though modern data acquisition systems already contain many features to reduce noise. The possible sources of noise are electrical noise, magnetic noise, electrostatic or capacitive noise, radio frequency (RF) noise [71]....etc. To reduce the noise to an acceptable

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Figure 5.3 Window interface control panel for creep data acquisition system

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level, both hardware techniques and software techniques have to be employed.

5.3.1 Hardware techniques

First of all, calibrate the data acquisition board before using. Keeping cables (wires) short and avoiding unnecessary connections (using one line rather than connecting two lines or more) are the primary principle that should be followed. Using a common ground for all equipment is also very important.

Electrostatic noise is produced by capacitive coupling between signal-carrying wires and other conductors or the ground. The most effective way to reduce this noise is to shield signal-carrying wires by wrapping them in a conductor that is grounded [71]. In most cases, the shield should be grounded at the signal source.

Separating signal-carrying wires and AC power lines can reduce magnetic noise. A high-pass filter is helpful to remove 60 Hz AC power interference which causes electrical noise.

Floating signal sources, *i.e.* strain gauge transducers, thermocouples, have isolated ground reference points. Coaxial cable should be used to make shielded connections to the floating signal source.

Mechanical vibration can also induce noise, especially for a strain gauge transducer. The sources of mechanical vibration include noisy machine with heavy vibration and pressure variations in water pipes used for cooling system.

In summary, short and well-shielded coaxial cable is good for signal-carrying and helps reduce noise. Use of a common ground for all equipment is necessary.

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5.3.2 Software techniques

If the noise is randomly distributed, signal averaging is one software technique that has been widely used to reduce noise. The standard deviation of signal average is inversely proportional to $(N)^{1/2}$ [71] where N is the number of data to be collected. Therefore, as more samples are taken, the standard deviation of signal average is reduced, and the average value becomes close to the true value. The signal-to-noise ratio is thus improved.

Curve-fitting is another way to approach the true value of those random data. The fitting function is chosen according to the shape of the signal waveform. As mentioned in Chapter III, the θ -projection constitutive equation is good for creep data analysis and was used for creep data analysis.

CHAPTER VI

EXPERIMENTAL PROCEDURES

6.1 SPECIMEN DESIGN

Fujiwara, Yamaguchi and colleagues [55,72] identified the room temperature deformation modes of PST crystal as {111}<112] twinning and {111}<110] normal dislocation slip. These twinning and slip systems are still operative at high temperatures, but some different results were found for the different test conditions. Soboyejo et al. [73] found the twinning activity is limited and slip involves 1/2 [110] unit dislocations and [101] and [112] superlattice dislocation in Ti-49Al-3.4Nb (at%) at 815 °C and 982 °C. However, Huang and Kim [11] found the 1/2 < 110 type dislocations in both gamma grains and gamma lamella in Ti-47Al-1Cr-1V-2.5Nb (at%) at 900 °C, but no superlattice dislocation and twin deformation were observed. On the other hand, Jin and Bieler [65,66] found a significant contribution of mechanical twinning to creep deformation at a relatively low temperature of 700 °C. As the temperature increases to 815 °C, the contribution of mechanical twinning to creep is significantly decreased. The reasons for these different results are not yet clear. The deformation conditions, chemical compositions and microstructure are believed to be some important factors which affect the deformation modes of creep in TiAl.

For lamellar microstructure, there are two deformation modes: the shear deformation across the lamellar boundaries (hard mode) and the shear deformation parallel to the lamellar boundaries (easy mode). Therefore, the deformation properties are affected

significantly by the orientation between the lamellar boundaries and the loading axis. Yamaguchi *et al.* [72] did a series of experiments on PST crystals in different orientations. Some specimens had lamellar boundaries oriented so that the deformation shear strains in the gamma lamellae were parallel to the lamellar boundaries, and other orientations required shear across the lamellar boundaries and lamellae of the α_2 phase. The relationship between the yield stress of PST crystal and the angle ϕ , which describes the angle between the lamellar boundaries and the loading axis, was plotted as shown in Figure 6.1 [72].

From Figure 6.1, we observe that the yield stress of TiAl is high when the lamellar boundaries are parallel or perpendicular to the loading axis, due to the difficulty of shear perpendicular to lamellae. On the other hand, for those angles $\phi = 30^{\circ} - 60^{\circ}$ orientations, the easy shear deformation which parallels to lamellae results in low yield stress value, especially in the maximum resolved shear stress orientation, $\phi = 45^{\circ}$.

In order to examine creep mechanisms under maximum resolved shear stress state, a compression fixture with the orientation of 45° to the tensile axis was used (see Figure 6.2). The compression test was used since it was more convenient than a tension test for a small specimen. By careful orientation, the {111} lamellar planes were parallel to the surface 45° from the loading axis and normal dislocation <110> directions were parallel to the principle resolved shear stress. Polycrystalline specimens were used for comparison. More experiments will be done on {111}<12] twinning-type deformation mode in future experiments by a colleague who is investigating this in more detail.



Figure 6.1 Plots of yield stress of PST TiAl in tension and compression as a function of the angle \$\oplus\$. The specimens were oriented so that the lamellar boundaries are perpendicular to the wider surface of specimens which is parallel to {112}(group 1) or {110}(group 2) [72]



Figure 6.2 Schematic of extensometer with compression-shear fixture and shape of specimen

6.2 MATERIAL AND SPECIMEN PREPARATION

Two types of TiAl were chosen for this research. One is polycrystalline which was made by Howmet Corp., Whitehall, MI. The fabrication process consists of a series of heat treatments which include hot isostatically pressed (HIPed) at 172 MPa and 1260 °C for 4 hours and then heat treated in Ar at 1300 °C for 20 hours and gas fan cooled at a cooling rate of 65 °C/min. The microstructure before creep deformation was duplex with $(\gamma+\alpha_2)$ lamellar grains plus equiaxed γ grains. The other type is a PST crystal which was grown in the LSRM facility at the University of Pennsylvania. The PST crystal was grown from the polycrystalline material using the ASGAL optical floating zone furnace in a flowing argon environment. The seed and feed was translated vertically through the hot zone to obtain a growth rate of 5 mm/hr. Both PST and polycrystal specimens have similar composition as shown in Table 6.1 [66].

Table 6.1 Chemical composition of Ti-47Al-2Cr-2Nb specimens [66]

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

Three specimens were made for three different compression creep tests. Two polycrystalline specimens were cut in the form of 5x5x3 mm bricks to fit the compressionshear fixture for testing in air or in flowing argon. One PST crystal specimen was cut in the

same form as polycrystalline specimen (5x5x3 mm brick), but the orientation was carefully determined by using a Laue camera and X-Ray diffraction so that the {111} lamellar plane was parallel to the 5x5 mm surface and the <110> direction was normal to one of the other surfaces. The compression extensometer with compression-shear fixture and the form of specimen are shown in Figure 6.2.

6.3 CREEP TEST SYSTEM

Three creep tests were done under compression by using an ATS series 2710 creep testing machine, and the creep data were recorded by the data acquisition system built for creep tests. The ATS system is designed to perform either creep testing (constant load) or stress relaxation testing (constant strain). The machine has a lever arm ratio of 20:1 to provide high load capacity and loading rate. The creep frame controller unit, which was made by ATS and Electronic Instrument Research Corp.(EIR), allows automatic control mode for constant load or stress creep test.

The strain was measured by Linear Variable Capacitance Transducers (LVCT) which perform precise and stable specimen gaging. A small change in length is measured as a change in capacitance. Two LVCTs were used so that if one fails there would be a backup. The LVCTs connect to the specially designed extensometer for compression creep test, and they directly measure the change in length during the creep test. The measurement from the LVCT and load cell are input into the control unit and show on a display panel. The control unit also provides the analog outputs of strain and load to allow the user to connect to the data acquisition system. The analog outputs are limited on -10~10V and the initial position of LVCT was adjusted to be within this limit.

The furnace has three heating zones that were controlled by a furnace control unit (LFE model 2010). The temperature can reach as high as 1200 °C and temperature gradient between top and bottom of the chamber can be controlled by careful adjustment of three heating zones. K-type thermocouples were used for measurement of temperature and were displayed on the panel of the furnace control unit. The furnace control unit also provides one analog output channel that was monitored by the data acquisition system.

The data acquisition system consists of IBM PS/2 286 computer and AT-MIO-16L-25 multifunction data acquisition board (for details see Chapter V). The five analog input channels were scanned and the values of load, displacements (A & B), temperatures (top and bottom of the specimen) were recorded. A sampling rate of 5000 pts/sec was used. The flow chart of the data acquisition program for creep test is shown in Figure 6.3 and program codes are shown in APPENDIX

Time-savings and precise data recording were obtained by using the data acquisition system. Since precise data are needed for creep tests, especially for stress and temperature change processes, the precision and noise are two issues that require more concern.

6.3.1 Precision

High resolution data are needed for the analysis of the stress and temperature change process. For example, in order to resolve elastic changes of a 3 mm tensile specimen of TiAl in a 10 MPa stress change, we need to record 1 data point for every 2 mV change (= 5.08×10^{-4} mm change in displacement) since the elastic changes is ~ 5.1×10^{-4} mm in this stress change. Rough data points will significantly influence the experimental result.



Figure 6.3 Flow chart of data acquisition program for creep test

The precision of the data acquisition board can be improved by increasing the gain. Table 5.1 shows the overall input range and precision according to the input range configuration and gain used. With the proper gain setting, the full resolution of the Analog/Digital Converter can be used to measure the input signal. For our AT-MIO-16L-25 board, the gain has four possible choices: 1, 10, 100 and 500. The highest precision of 4.88 μ V can be obtained by using the combination of 0 to 10 V input range and gain of 500.

In our application, the combination of -10V to 10V input range and gain of 1 was used because the -10 to 10 V input range is wide enough for measurement of larger strain and easy for setting the position of LVCT. However, the low precision of 4.88 mV caused some inaccuracies during the recording of stress and temperature change process data. The gain of 10 with higher precision (488 μ V) may account for this problem. However, the narrower input range of -1 to 1V for a gain of 10 was not convenient for long-term creep test since only 2V (0.508 mm) range is allowed for LVCT to monitor the heat-up process and measure the strain. The accommodation between the wide input range (enough range for measurement of large strain) and high precision (enough for measurement of small voltage changes) requires practical compromise.

6.3.2 Noise

In order to minimize the noise, the both hardware and software techniques should be used.

(a) Hardware

In general, acceptable operation of the test system in test control and data

acquisition depends on the minimization of noise pickup, primarily 60 Hz. A common grounding system for all devices was implemented. The LVCTs were also grounded through the connection to the control unit of the ATS machine. Since the LVCTs were cooled by the cooling system, the temperature-induced noise was avoided. Furthermore, the extensometer was electrically isolated from the specimen and the specimen grips, and also from the load frame. The common grounding is a very important procedure to minimize the noise in the data.

By the recommendation of the AT-MIO-16 User Manual, individually shielded, twisted-pair wires were used to connect analog input signals to the AT-MIO-16 board. Differential analog input connections were also used to reject the possible noise in the common ground mode. By the way, carefully separated the signal lines from high-current or high-voltage lines to reduce the magnetic-induced noise. Another possible source of noise in our creep tests is the mechanical vibration due to a noisy machine that generates heavy vibration in the laboratory from time to time.

The calibration of the data acquisition system is helpful to acquire precise data. For analog input calibration, the measurement accuracy within $\pm 1/2$ least significant bit (LSB) is recommended by the AT-MIO-16 User Manual [74]. The LSB of the input range we used ($-10 \sim +10V$) is 4.88 mV and measurement accuracy should be within ± 2.44 mV. A constant was added to the signal obtained from the data acquisition board to bring the DAS value within ± 1 mV of the value obtained from the LVCT.

Actually, the biggest unexpected variation of displacement fluctuation is due to the loading compensation feedback system of the ATS machine. The constant load was maintained by the ATS control unit and a slow moving DC motor adjusted the tension on

the loading arm to maintain a constant load. The window of the feedback loop was within $\pm 1\%$. The measured value of displacement fluctuated as the load increased or decreased to maintain the constant-load value. In the way of a hardware technique, narrowing the load range between high and low limits will lower the fluctuation on loading and displacement.

(b) Software

Using software techniques, the signal-averaging method was used to reduce the noise caused by fluctuation of loading or other sources. By sampling at a higher rate and using the signal-averaging method, it is a possible to minimize the errors. In our application, the sampling rate of 5000 pts/sec for 5 channels, *i.e.* 1000 pts/sec per channel, was used. Thus, each data point represents an arithmetic mean of 1000 data points sampled at 1 kHz frequency.

Each data point was evaluated with respect to a change criteria called VINC, which is calculated by the equation:

$$VINC = FS * LO * 10000(mV) / 0.1(in) / N(pts)$$
(24)

where FS is the final strain, LO is the initial length of specimen and N is the number of points to be recorded. For example, if the final strain of a creep test is 10% (0.1) and initial length of specimen is 0.1 in, and if 250 points data are expected to be recorded during this creep deformation process up to 0.1 strain, data should be taken and stored for every 4 mV change according to equation (24).

The noise effect caused by fluctuation of loading or other noise will influence the

shape of creep curve, as Figure 6.4 shows. The fluctuation value between high and low limits can also be averaged by using a signal-averaging method. A data value was temporarily stored in a circular buffer if this value was greater than the value of the previous value (V_i) +VINC. After 20 data points were stored in the buffer, the average of those 20 points was calculated and compared with the value of V_i +VINC. If the average of 20 points was greater than V_i +VINC, this average was stored. If not, no data point would be taken until the 20 points average is greater than V_i +VINC.

Since the window interface operation panel was created, the complicated computing processes slowed the performance of the computer. This problem is not serious during the constant load and temperature creep condition, but it is a very serious problem during the stress or temperature change process due to the high frequency of recording data that is necessary under this condition. To solve this problem, the data was stored to the memory temporarily during the stress and temperature change processes and after that it was written to disk.

6.4 CREEP TEST PROCEDURES

Three compression creep tests were done in air or Ar over the temperature range 760 $^{\circ}$ C ~ 810 $^{\circ}$ C. A series of stress and/or temperature changes were conducted for each test (see Table 6.2). In the beginning, the specimen was initially heated up to 760 $^{\circ}$ C step by step (~100 $^{\circ}$ C/hr) and the temperature difference between specimen top and bottom was controlled in 10 $^{\circ}$ C. The thermal expansion was compensated by using draw head adjustment so that constant load was maintained during the heat-up process. The



Figure 6.4 The creep curve varies with the fluctuating data.

Specimen	Polycrystalline-1	Polycrystalline-2	PST		
Microstructure	Duplex	Duplex	Fully lamellar		
Test temperature	764~775 ⁰C	765~800 °C	760~806 ^o C		
Test stress	68.8 ~87.8 MPa	75.7~82 MPa	19~31 MPa		
Final strain	0.274	0.457	0.068		
Test atmosphere	air	Ar	Ar		

Table 6.2 The conditions for each test

specimens were initially loaded slowly at a strain rate on the order of 10^{-4} S⁻¹ at 760 °C and a defined primary stage followed by a minimum strain rate region was obtained. Following a period well into the minimum strain rate region, the stress or temperature was increased from this level to the next level. The response to the change exhibited a transient decelerating strain rate followed by a minimum strain rate. Following a stress or temperature decrease, the response exhibited strain contraction followed by an accelerating strain rate until a nearly steady value was reached. This process was repeated for each stress and temperature level applied. Top and bottom sample temperatures were held within $\pm 1^{\circ}$ C.

The values of specimen load, displacement, temperature and elapsed time of the experiment were digitally recorded by computer and saved as data file in ASCII format. Assuming perfect shear deformation, i.e., no compression strain $\gamma = (2)^{1/2} \Delta l/t$, where Δl (mm)= 2.54x10⁻⁴ ΔV (mV) and t=3 mm, the shear stress and strain data were determined using spreadsheet programs. The creep data was analyzed by using the curve fitting software (TableCurve 3.11) with a modified creep equation of the θ -projection concept.

CHAPTER VII

RESULTS

The experimental results are shown in Figures 7.1-7.3. Every data point shown on the plots represents the average of 20 points calculated by the method described in the section 6.3.2. The resolution of 4.88 mV(= 1.24×10^{-3} mm) was used for the creep tests and the error of data was $\pm 0.124\%$. The lines in the data come from a curve fitting procedure by using the modified creep equation of the θ -projection concept described in section 3.4. The fitting parameters for PST and polycrystal in Ar and air are shown in Tables 7.1-7.3 and the fit standard errors are $3\sim5\times10^{-4}$.

"Normal" transient behaviors were observed for the PST and polycrystal specimens (in Ar or air) after stress or temperature change. As the stress or temperature increases, an instantaneous extension is followed by creep which decelerates toward the new (higher) steady state. On the other hand, as the stress or temperature decreases, an instantaneous contraction is followed by transient creep which accelerates to a new (lower) steady state.



Figure 7.1 The creep curve of PST (with fitting) (a) strain-time curve (b) strain-strain rate curve



Figure 7.2 The creep curve of polycrystal in air (with fitting) (a) strain-time curve (b) strain-strain rate curve



Figure 7.3 The creep curve of polycrystal in Ar (with fitting) (a) strain-time curve (b) strain-strain rate curve

stage	Conditions	ε ₀	А	α	έ _s	t ₀ (sec)	strain	Fit standard error
1	19.0 MPa 760 ºC	8.16 E-04	6.05 E-03	5.10 E-05	5.37 E-08	0	0.0123	5.33E-04
2	22.0 MPa 760 ºC	1.44 E-02	1.55 E-03	1.54 E-04	8.03 E-08	120420	0.0220	4.95E-04
3	25.3 MPa 760 °C	1.65 E-02	7.01 E-03	2.04 E-04	8.23 E-08	212544	0.0306	3.86E-04
4	25.1 MPa 773 ^o C	3.35 E-02	4.50 E-04	4.40 E-04	7.73 E-08	301320	0.0378	5.20E-04
5	25.1 MPa 760 ^o C	4.22 E-02	4.52 E-03	3.47 E-04	5.21 E-08	352620	0.0412	5.38E-04
6	25.0 MPa 790 °C	4.28 E-02	4.60 E-04	5.83 E-04	1.20 E-07	423144	0.0482	4.70E-04
7	25.1 MPa 761 ⁰ C	4.80 E-02	2.51 E-04	1.04 E-04	4.80 E-08	471492	0.0498	3.74E-04
8	25.1 MPa 806 ⁰ C	5.38 E-02	4.93 E-04	7.30 E-04	1.52 E-07	537048	0.0588	5.84E-04
9	25.1 MPa 766 ^o C	5.98 E-02	1.91 E-04	1.38 E-04	3.04 E-08	597147	0.0618	2.01E-04
10	31 MPa 762 ⁰ C	6.15 E-02	4.53 E-03	7.50 E-04	9.51 E-08	705708	0.0675	2.36E-04
11	25 MPa 763 ^o C	6.68 E-02	2.50 E-04	1.14 E-04	3.80 E-08	740736	0.0681	5.52E-04

Table 7.1 The fitting parameters for PST specimen in Ar

stage	Conditions	ε ₀	А	α	έ _s	t ₀ (sec)	strain	Fit standard error
1	75.7 MPa 764 ºC	3.92 E-03	1.89 E-02	2.41 E-05	1.14 E-07	0	0.1076	1.17E-03
2	87.8 MPa 764 ºC	1.17 E-01	2.20 E-03	6.87 E-05	1.87 E-07	767160	0.1564	4.52E-04
3	75.2 MPa 764 ºC	1.65 E-01	1.50 E-03	2.23 E-05	1.28 E-07	1022760	0.1854	2.76E-04
4	75.1 MPa 774 ºC	1.84 E-01	1.00 E-03	8.30 E-05	2.36 E-07	1186560	0.2633	4.66E-04
5	68.8 MPa 775 ⁰ C	2.60 E-01	4.97 E-03	3.12 E-05	1.95 E-07	1533960	0.2742	2.30E-04

Table 7.2 The fitting parameter for Polycrystal specimen in air

stage	Conditions	ε ₀	A	α	έ _s	t ₀ (sec)	strain	Fit standard error
1	75.7 MPa 765 ºC	2.89 E-03	3.94 E-02	5.19 E-05	3.21 E-07	0	0.0993	8.72E-04
2	75.7 МРа 770 °С	1.00 E-01	1.97 E-03	3.81 E-04	3.25 E-07	147276	0.1301	2.30E-04
3	82.0 MPa 770 ℃	1.36 E-01	2.73 E-03	2.08 E-04	3.84 E-07	233784	0.1684	1.03E-03
4	75.7 МРа 770 ^о С	1.64 E-01	1.00 E-03	1.11 E-04	2.66 E-07	301680	0.1848	2.41E-04
5	75.7 MPa 780 ℃	1.86 E-01	1.69 E-03	2.57 E-04	4.44 E-07	386424	0.2296	3.95E-04
6	75.7 MPa 770 ℃	2.33 E-01	1.02 E-03	9.43 E-05	2.38 E-07	483732	0.2925	2.18E-04
7	75.7 MPa 790 ℃	2.94 E-01	2.90 E-03	7.43 E-04	5.62 E-07	745560	0.3054	3.39E-04
8	75.7 MPa 770 ℃	3.12 E-01	1.01 E-03	7.43 E-05	2.66 E-07	771696	0.3220	9.18E-04
9	82.0 MPa 770 °C	3.31 E-01	1.26 E-03	1.84 E-04	2.87 E-07	849456	0.3544	9.87E-04
10	75.7 MPa 770 °C	3.53 E-01	1.03 E-03	4.84 E-05	2.32 E-07	925920	0.3679	****
11	75.7 MPa 800 °C	3.70 E-01	3.40 E-03	9.84 E-04	7.63 E-07	1001412	0.4361	1.16E-03
12	75.7 MPa 770 ^o C	4.35 E-01	3.03 E-03	9.84 E-05	4.02 E-07	1083816	0.4576	4.00E-04

Table 7.3 The fitting parameters for Polycrystal specimen in Ar

*: Too few data to fit

CHAPTER VIII

ANALYSIS AND DISCUSSION

8.1 CREEP BEHAVIOR

The creep curves of PST and polycrystalline specimens with stress and temperature change are shown on the Figures 7.1-7.3. As the stress or temperature increases, an instantaneous extension is followed by creep which decelerates toward the new (higher) steady state rate. On the other hand, as the stress or temperature decreases, an instantaneous contraction is followed by transient creep which accelerates to a new (lower) steady state. This is a typical form of "normal" transient behavior and consistent with the TEM observation of dislocation network observed by Jin *et al.* [67]. Jin *et al.* [67] found that the creep deformation mode in TiAl is pure metal type (Class M) and subgrain boundaries were observed both in equiaxed gamma grains and within gamma laths in lamellar grains in the creep deformed specimen of duplex structure.

Since there is no long term steady state creep rate in TiAl, the minimum creep rate is used as a basis for comparison for the power law equation. The values of minimum creep rate were obtained by fitting the modified θ -projection equations (Equation 16,17), and the minimum strain rate as usually used to represent the steady state creep rate in the θ -projection equation. In the θ -projection equation, the minimum creep rate represents a balance between the hardening term and softening term. This is similar to the dislocation theory of steady state deformation presented by Gottstein and Argon [75]. According to their model, a master equation based on the accepted dislocation theory, considering various recovery mechanisms, was derived. This equation can account for creep and CSR (constant strain rate) tests with respect to compatibility, transients and steady state deformation because both modes of deformation have similar microstructure development.

8.1.1 Stress exponent

<u>(a)PST</u>

As there is no deformation mechanism map for TiAl available in the literature, and as the creep behavior of TiAl is closer to FCC Al than to HCP Ti for the temperature examined, the creep deformation map for pure Al was considered (see Figure 8.1).

For the stress and temperature regime examined (T~1/2 T_m =760 °C, σ ~19-31 MPa), the normalized shear stress (σ /G) was in the high temperature creep regime for both 10µm and 1mm grain size Al (G=(73-1.2 x 10⁻²T/K) GPa) [68], *i.e.* the power-law creep by dislocation glide-plus-climb. This dislocation glide-plus-climb process is limited by the lattice-diffusion controlled climb.

According to the power law equation (Equation 1), at each temperature, the stress exponent is given by the gradient of the $\log \varepsilon_s$ /logo plot. This gradient is shown for each stress change as the experiment progressed, as indicated by the dashed lines in Figure 8.2. Thus, by comparing the minimum creep rates following stress changes in the strain range of 1%~3% and 6%~7%, the stress exponents n=3 and n=5.6 were obtained, respectively (see Figure 8.2).

The stress exponent of n=3 is characteristic of dislocation glide creep and n=5 for dislocation climb creep. There are two possible ways to explain stress exponent of n=3, one is no steady state was reached in the strain range of $1\% \sim 3\%$, so, this value can be



(a)



(b)

Figure 8.1 The deformation mechanism maps of pure aluminium. (a) grain size 10 µm (b) grain size 1 mm [76]





Figure 8.2 Plots of stress dependence of minimum creep rate for PST and polycrystal. The dash lines indicate the history of deformation. #'s next to datum points refer to Tables 7.1-7.3.

doubted. On the other hand, the pipe diffusion did not contribute to creep deformation in the early stage (1%~3% strain) since fewer dislocations. In the latter stage (6%~7% strain), more dislocations lead larger contributions of pipe diffusion and cause the stress exponent of n=3 increased by 2.

(b) Polycrystal

For polycrystal in air, the stress exponents of n=2 for 774 ^oC and n=3 for 764 ^oC were obtained (see Figure 8.2). The position of the normalized stress and temperature on the mechanism boundary of the deformation mechanism map for Al, suggests that for this temperature regime, there is no single dominant deformation mechanism, and lies near the dislocation climb controlled and dislocation glide controlled creep boundary.

The stress exponent n=3 is commonly associated with the dislocation glide-limited creep process. But dislocation glide-limited deformation usually causes the "inverse" transient behavior after stress or temperature changes rather than normal transients we observed. One possible interpretation for n=3 is Coble creep (n=1) modified by the pipe diffusion which usually increases the stress exponent by 2 [8]. However, from the deformation mechanism map in Figure 8.1(a)(b), the applied stress of 75~87 MPa seems too high for Coble creep to be the dominant mechanism. According to the Coble creep equation [25],

$$\dot{\varepsilon} = \frac{B_c}{\pi} \frac{D_b \delta_b \sigma \Omega}{d^3 kT}$$
(25)

where $\dot{\epsilon}$ is the Coble creep rate, D_b is the grain boundary diffusion coefficient, δ_b is the effective grain boundary width, Ω is the atomic volume, d is the grain size and B_c is the

constant (=148). Grain sizes for Coble creep were estimated and shown on the Table 8.1 using the range data for FCC metal of Frost and Ashby [76]. Since only small volume fraction of α_2 phase is presented, only γ phase was considered to estimate the grain size for Coble creep. Critical grain size range of 10 μ m~28 μ m for PST, and 12 μ m~34 μ m for polycrystal were calculated for Coble creep, which are smaller than the average measured grain size (>50 μ m) of polycrystal specimen we used. Therefore, if Coble creep occurs, then it must occur in lamellar microstructure regions, which have sizes <~10 μ m.

Since the test conditions of the polycrystal may lie in the transition regime of different mechanisms, the amount of the contribution of each deformation mechanism to the creep deformation becomes a more complicated problem, especially since the microstructure also plays a important role for interpretation of creep behavior. Therefore, more experiments in a wider test conditions are necessary to gain better understanding and interpretation of those unsolved problems.

For polycrystal in Ar, the stress exponent $n=2\sim3$ was calculated at 770 °C (see Figure 8.2). It is similar to that of polycrystal in air and there seems to be no significant influence on the stress exponent by different atmospheres (air or Ar).

8.1.2 Activation energy

<u>(a)PST</u>

The activation energy $Q_c = 137 \pm 70$ KJ/mole was obtained for PST in the temperature range of 760 °C~806 °C (see Figure 8.3). This value is similar to the activation energy Q=150 KJ/mole for interdiffusion in γ -TiAl which was reported by Ouchi *et al*. [64]. The lower activation energy maybe due to the easy mode deformation. A similar

Table 8.1 The grain sizes of PST and polycrystal for Coble creep

	PST	polycrystal
Applied stress σ (MPa)	22	88
Steady state strain rate (s ⁻¹)	8.23E-8	1.87E-7
Temperature (K)	1033	1037
Grain size for Coble creep(µm)	10~28	12~34

- * $Q_b = 150$ KJ/mole was assumed.
- * δD_b for FCC metals: 9.1x10⁻²³~2.1x10⁻²¹ m³S⁻¹
- * $\Omega = 1.18 \times 10^{-29} \text{ m}^3 \text{ for TiAl}$

activation energy value of 130 KJ/mole for thermally activated glide of screw dislocation in γ -TiAl at room temperature was reported by Appel *et al.* [77].

(b)Polycrystal

The activation energy of 574 ± 90 KJ/mole and 229 ± 60 KJ/mole were computed for air and Ar atmosphere, respectively (see Figure 8.3). The value of 229 ± 60 KJ/mole (in Ar atmosphere) is lower than the general accepted value Q =291 KJ/mole for Ti diffusion in Ti-54Al which was reported by Kroll *et al.* [60]. On the other hand, a high activation energy Q_C =574 ± 90 KJ/mole was obtained in air and it is not common for stress exponent of n=2~3. This value is unreliable since only two temperature changes. Dynamic recrystallization has been reported [8] to be a important phenomenon at higher temperature (~760 0 C) and at strains > ~ 5% it may cause this high activation energy for the polycrystal in air. However, this unsolved problem needs more experiments in the wider range of temperature and stress since Q_C=574 ± 90 KJ/mole for the polycrystal in air was determined by interpreting only two data points of different temperatures. Since the uncertainty of activation energy for polycrystal in air, it is not known about the influence of atmosphere (Ar and air) by the comparison of activation energy.

8.1.3 Stress and temperature change

In general, the stress change experiment is usually used to study the substructure phenomenon during the creep deformation since it is easy to conduct. The temperature change experiment is seldom used because it is not easy to control the temperature fluctuation after temperature changes. From our observations after temperature changes, the



Figure 8.3 Arrhenius plots of the temperature dependence of minimum creep rate for PST and polycrystal of TiAl. The dash lines indicate the history of deformation. #'s next to datum points refer to Tables 7.1-7.3

temperature generally fluctuated in the range of T_c (creep temperature) \pm 5-7 ° C for half hour before T_c settled down. This feature may result in some uncertainties in the interpretations of transient behaviors. However, normal transients were still observed after temperature changes as well as stress changes. This suggests that stress and temperature have similar influence on the substructure during the creep deformation.

From the loading history plots in Figure 8.2, 8.3, as stress or temperature increases, the creep rate $\dot{\epsilon}_m$ increases to a new value (> $\dot{\epsilon}_m$) and then back to a lower value (< $\dot{\epsilon}_m$) after stress or temperature reduction. This process can result from subgrains that were formed by dislocation climb processes that harden the specimen in the primary stage. These substructures in primary creep are evident in the results of Jin *et al.* [67] and indicated that the creep behavior in TiAl is Class M type behavior.

8.1.4 Composite model

Since the mechanisms of creep of in TiAl are complicated, the composite model has been presented by several researcher [10,68]. Those composite models may provide a possible way to understand those unsolved mysteries of the creep deformation in polycrystal. The composite model uses a simple rule-of-mixture equation to combine all deformation mechanisms which contribute the creep deformation. This is a useful method to describe creep behaviors which have more than one deformation mechanism. A less sophisticated, empirical model and analysis is explored next,

$$\dot{\varepsilon}_{c} = \sum_{i} V \dot{\varepsilon}_{i}$$
(26)

 $\dot{\mathbf{e}}_{c}$ is the total creep rate, $\dot{\mathbf{e}}_{i}$ is the creep rate of mechanism i, V_{i} is the volume fraction dominated by mechanism i. The analysis needed to identify the operative mechanism is beyond the scope of this thesis.

8.2 IMPLICATIONS OF THE FITTING PARAMETERS

A modified θ -projection concept equation [39] was used for the data analysis of creep to explore the creep behavior. The constitutive equation for transient creep after stress and temperature increase is

$$\varepsilon = \varepsilon_0 + A \left\{ 1 - \exp\left(-\alpha \left(t - t_0\right)\right) \right\} + \dot{\varepsilon}_s \left(t - t_0\right)$$
(16)

where ε_0 is an adjustable parameter, t_0 is starting time. $\dot{\varepsilon}_s$ is the steady state (or minimum) strain rate, A and α are curve fitting parameters. In the case of transient creep after stress and temperature decrease, the constitutive equation is

$$\varepsilon = \varepsilon_0 + A \exp\left(-\alpha \left(t - t_0\right)\right) + \dot{\varepsilon}_s \left(t - t_0\right)$$
(17)

8.2.1. Parameter A (strain hardening parameter)

The strain hardening parameter A represents the saturation strain of primary creep. The parameters A are plotted against σ/G in Figure 8.4(a) and show that the parameter A depends on stress. The values of A increase as the applied shear stress increases for PST and polycrystal specimens. This is consistent with the results found on CrMoV steels by Maruyama and Oikawa [45] which presented A = f_a(σ/E), where f_a(σ/E) is a function only of σ/E (see Figure 8.4(b)). These results also suggest that the stress increase was associated with the microstructure change.

The values of A are also plotted against the temperature, as shown in Figure 8.5. Strain hardening parameter A is independent of temperature for the PST. On the other hand, no matter what atmosphere (Ar or air), the values of A of polycrystal appear to increase as temperature increases. It indicated that the microstructure of polycrystal is more harden with temperature increase than PST in the temperature range of 760 $^{\circ}$ C ~ 800 $^{\circ}$ C.

8.2.2. Parameter α (rate constant)

The stress and temperature dependence of the rate constant α for the PST and polycrystal are shown in Figures 8.6(a), 8.7(a), respectively, and show the parameter α increases as stress or temperature increases. It is consistent with other observations using the θ -projection concept (see Figure 8.6(b), 8.7 (b)). Those studies of the θ -projection concept [39,43~45] suggest that α can be expressed in the form of

$$\alpha = f_{\alpha}(\sigma/E)D \text{ where } D = D_0 \exp(-Q_D/RT)$$
(10)

where D is the self-diffusion coefficient and Q_D is the activation energy for the rate constant.

Brown *et al.* [44] considered that α has the common activation energy as the self diffusion. Maruyama *et.al.* [79] stated that in the case of dislocation creep controlled by diffusion, the rate constant α of TiAl can be formed as $\alpha = CD\sigma^{m+2}$, where C is a constant, D is the diffusion coefficient, m is the effective stress exponent for dislocation velocity and the stress exponent of 2 comes from the stress dependence of dislocation



Figure 8.4 Stress dependence of strain hardening parameters A.

- (a) PST and polycrystal of TiAl
- (b) CrMoV steel by K. Maruyama et al. [45]
- #'s next to datum points refer to Tables 7.1-7.3


Figure 8.5 Plots of the strain hardening parameters A against temperature for PST and polycrystal of TiAl. #'s next to datum points refer to Tables 7.1-7.3



(a)



(b)



(a) PST and polycrystal of TiAl

(b) Ti-50Al and Ti-51Al by K. Maruyama et al.[79]

#'s next to datum points refer to Tables 7.1-7.3.







Figure 8.7 The temperature dependence of the rate constants α (a) PST and polycrystal of TiAl

- (b) CrMoV steel by K. Maruyama et al. [45]
- #'s next to datum points refer to Tables 7.1-7.3.

density. Some reports [8,75] have shown that the stress exponent can be increased by a factor of 2 when pipe diffusion is rate-controlling during intermediate temperature creep deformation.

From the stress dependence of the rate constant plot in Figure 8.6 (a), the slopes n = 5.5 for PST and n = 3 for polycrystal are close to the stress exponents which were obtained for steady state creep (n=5.6 for the PST and n=2-3 for the polycrystal in Ar). Figure 8.6(b) also shows similar result of slope n-5.6 in Ti-50Al and Ti-50Al by K. Maruyama *et al.* [79]. However, these stress exponents in Figure 8.6(a)(b) include a factor of 2 which is contributed from pipe (core) diffusion. Therefore, the n=5.5 suggests dislocation glide (n-3) aided by pipe diffusion to be the dominant deformation mechanism in PST since higher dislocation density causes the significant contribution of the pipe diffusion. On the other hand, dynamic recrystallization that occurs in polycrystal results in fewer dislocations and consequently less pipe diffusion, and so the n=3 was obtained. The high stress exponent of n=7 for the polycrystal in air is much higher than the stress exponent n=2-3 which was obtained for steady state creep. This may be due to a slope with error resulting from only two points or a curve fit for one of these is incorrect.

The activation energies Q_D of the rate constant for PST and polycrystal were calculated by the Arrhenius plots of the temperature dependence of α , and the $Q_D = 133 \pm 20$ KJ/mole for PST, $Q_D = 294 \pm 60$ KJ/mole for polycrystal in Ar and $Q_D = 412 \pm 90$ KJ/mole for polycrystal in air were given, respectively (see Figure 8.7).

For the PST crystal, the value of $Q_D = 133 \pm 20$ KJ/mole is close to the apparent activation energy of creep, $Q_C=137 \pm 70$ KJ./mole, calculated from the temperature dependence of minimum creep rate. The interpretation of the activation energy for the rate

constant provides that the same rate-limiting thermal activation process occurs in transient steady state creep. Therefore, the activation energy $Q_C=135 \pm 70$ KJ/mole for the easy deformation mode creep of PST should be a reasonable interpretation.

For the polycrystal in Ar, the activation energy $Q_D = 294 \pm 60$ KJ/mole for rate constant is higher than activation energy $Q_C=229 \pm 60$ KJ/mole which was obtained from the Arrehenius plot of temperature dependence of minimum creep rate. The $Q_D = 294 \pm 60$ KJ/mole is closer to a general accepted activation energy value (Q = 291KJ/mole) for gamma TiAl in this temperature and stress regime [60]. However, plots of temperaturecompensated creep rate (Zener-Hollomon parameter Z = $\dot{\epsilon}_s \exp(Q_c /RT)$ [19] in Figure 8.8 shows that the lower activation energy causes better superimposition data onto a single line. It implies that the Q_C=229 KJ/mole or 150 KJ/mole better describes this creep deformation process.

From the interpretation of steady state creep rate and the fitting parameters of θ projection equation, the stress exponent of n=5.6 for PST appears to be due to the dislocation glide (n~3) aided by pipe diffusion. The occurrence of pipe diffusion is supported by
the low activation energy Q_C=135 ± 70 KJ./mole. For the polycrystal, fewer dislocations
existed due to dynamic recrystallization, this can account for the stress exponent of n~3
and high activation energy of Q_C=229 ± 60 KJ/mole (in Ar) and Q_C=574 ± 90 KJ/mole
(in air). Even though dislocation glide (n~3) usually causes "inverse" transient behavior,
the n~3 is still acceptable for the "normal" transient observations in PST and polycrystal.
Weertman and Weertman [80], who have shown that, when glide and climb distances
(velocities) are similar, dislocation climb can occur with n=3 rate law. Similar result also
was found by Gottstein and Argon [75]. According to their dislocation theory model, the



Figure 8.8 Plots of temperature-compensated creep rate for polycrystal in Ar

stress exponent of n=3 was found when only recovery by climb is taken into account.

Finally, from the interpretation of stress exponent and activation energy, we found the quality of data in PST appears better than in polycrystal in spite of the fact of that fewer data in PST causes greater uncertainties in curve fit. That indicated the simpler deformation process occurs in the PST and that the PST data are sufficient reliable.

CHAPTER IX

CONCLUSION

The automated data acquisition system which consists of PC-based computer and data acquisition board was developed for transient creep test. The purpose of time-savings was achieved and accurate data were obtained for transient creep test. The effect of noise was minimized by using hardware (common ground) and software (signal-averaging) techniques.

'Normal' transient creep after stress and temperature change were observed on the PST and polycrystal (duplex) specimens. This suggests that Class M type creep deformation and subgrain structures were formed during the primary creep stage.

The stress exponent of n=5.6 at 25 ~31 MPa and activation energy Q= 135 ± 70 KJ/mole at 760 ~806 0 C for the PST suggest that the dislocation glide aided by pipe diffusion is the dominant mechanism of creep deformation in this microstructure condition. For the polycrystal, the stress exponent n=2~3 and the activation energies of Q= 229 ± 60 KJ/mole (in Ar), Q= 574 ± 90 KJ/mole (in air) were found. The high activation energy and low stress exponent suggests that dynamic recrystallization occurs that the dislocation network forms, due to the normal transient, but glide is limiting. Creep characteristics of polycrystal are affected by dynamic recrystallization could cause the complicated creep behaviors which were observed. A composite model can account for multiple deformation mechanisms is suggested for interpretation of this kind of complicated creep behavior.

The data analysis was aided by the modified θ -projection equations for creep. The

implication of fitting parameters of θ -projection equation provide a remarkable information to interpret the complicated creep behavior in TiAl. APPENDIX

APPENDIX

PROGRAM CODES FOR CREEP TEST

REM \$INCLUDE: 'creep.inc'

REM ************************************	**
REM * some important parameters:	*
REM *	*
REM * VL : the load	*
REM * VSA : the strain of channel A(min volts)	*
REM * VSB : the strain of channel B(min volts)	*
REM * TC1 : the temperature of specimen top	*
REM * TC2 : the temperature of specimen bottom	*
REM * FS : the final strain	*
REM * FT : the final time	*
REM * L0 : the initial specimen length	*
REM * A0 : the initial specimen area	*
REM * ID : the specimen ID	*
REM *	*
REM ************************************	**
REM	
DECLARE SUB get.data(VL,VSA,VSB,TC1,TC2)	
DECLARE SUB write.data(FILENAME\$)	
DECLARE SUB average.data(Iavg%,VAVG,CTIME,VL,VSA,VSB,	ГС1)
REM declare the parameters ************************************	******
DEFDBL A-Z	
COMMON SHARED /data.buf/data.buf%()	
COMMON SHARED /chans/chans%()	
COMMON SHARED /gains/gains%()	
COMMON SHARED /volts.buf/volts.buf#()	
COMMON SHARED /CDATA/CDATA#()	
COMMON SHARED /AVG/AVG#()	
COMMON SHARED /NUMPOINTS/NUMPOINTS AS INTEGER	
COMMON SHARED /RATE/RATE AS DOUBLE	
COMMON SHARED /VSB0/VSB0 AS DOUBLE	
COMMON SHARED /dspace/dspace as string	
COMMON SHARED /Ikeep/Ikeep as integer	
COMMON SHARED /X/X#()	
COMMON SHARED /Y/Y#()	

COMMON SHARED /hPanel/hPanel as integer COMMON SHARED /count/count as integer COMMON SHARED /handle/handle as integer COMMON SHARED /Ifull/Ifull as integer CONST NUMCHANS% = 5' the # of input channels **REM define data buffers for storing** ' buffer for storing binary data DIM data.buf%(5000) ' buffer for storing voltage data DIM volts.buf#(5000) ' array storing channels DIM chans%(NUMCHANS%) ' array storing gains DIM gains%(NUMCHANS%) '6 channels of averaged data DIM CDATA#(NUMCHANS%,500) DIM AVG#(4,20) ' array to average 10 pts ' the string storing current date **DIM NOWDATE AS STRING * 20** ' the output filename **DIM FILENAME AS STRING * 81** ' the string buffer DIM buf as string * 80 DIM STARTDATE as string * 15 ' the start date **DIM STARTIME** as string * 10 ' the start time DIM scanarray#(10) ' the arrry for the data scanning from buf\$ DIM X#(2000) ' x-axis graph data(time) 'y-axis graph data(strain) DIM Y#(2000) i% = 0' loop counter and array index k..err% = 0' holds error code quitloop1% = 0' loop control flag#1 ' loop control flag#2 quitloop2% = 0quitloop3% = 0' loop control flag#3 hPanel% = 0' creep test panel handler ' VarChange panel handler varPanel% = 0 $\operatorname{ctrl}\% = 0$ ' control ID numTimeOutTicks& = 0' tick count for Timeout_Config '# of days of the experiment period NDAYS% = 0**PTSPERCHAN%** = 200' the # of data to acquire per channel NUMPOINTS% = NUMCHANS%*PTSPERCHAN% 'total number of points' to

NUMPOINTS% = NUMCHANS%*PTSPERCHAN% ' total number of points ' to 'acquire RATE = 5000# ' sampling rate

PRINT "Current acquisition parameters are: " PRINT NUMCHANS%; "channels" PRINT PTSPERCHAN%; "pts/chan" PRINT RATE; "points/sec"

```
WHILE i% < NUMCHANS%
 chans\%(i\%) = i\%
 gains\%(i\%) = 1
 i\% = i\% + 1
WEND
REM initializing the AT-MIO-16 Board
k..err% = Init.DA.Brds%(1, boardType%)
k..err% = OpenInterfaceManager%
                           'open the interface
numTimeOutTicks& = (NUMPOINTS% / RATE) * 20
IF numTimeOutTicks& < 20 THEN
 numTimeOutTicks\& = 20
END IF
k..err% = Timeout.Config%(1, numTimeOutTicks&)
k..err% = FileSelectPopup("","*.dat","The Output Filename ",0,0,1,FILENAME$)
SELECT CASE k..err%
 CASE 0
 k..err% = FileSelectPopup("","*.dat", "The Output Filename ",0,0,1,
                     FILENAME$)
 CASE 1
  quitloop2\% = 1
 CASE 2
  quitloop2\% = 0
 CASE ELSE
  STOP
END SELECT
hPanel% = LoadPanel% ("creep.uir", CT%)
varPanel% = LoadPanel%("creep.uir",VC%)
```

```
k..err% = DisplayPanel% (hPanel%)
```

```
k..err\% = HidePanel\%(varPanel\%)
i..dummy% = SetCtrlVal%(hPanel%, CT.ppc%, PTSPERCHAN%)
i..dummy% = SetCtrlVal%(hPanel%, CT.rate%, RATE)
dspace$ = ", "
IF quitloop2\% = 1 THEN
REM the continuing experiment
handle%=OpenFile(FILENAME$,0, 1, 1)
 IF handle\% = -1 THEN
 i..dummy% = SetCtrlVal%(hPanel%,CT.mw%,"error opening file")
 STOP
 ELSE
 END IF
 position \& = SetFilePtr(handle\%, 0, 0)
 n\% = Readline(handle\%,buf\$,79)
 print buf$
 n\% = Scan (buf\$, "\%s>\%5f[x]", scanarray#())
 L0 = scanarray #(0)
                          'specimen length
                          'specimen area
 A0 = scanarray#(1)
 FS = scanarray#(2)
                          'estimated final strain
 FT = scanarrav#(3)
                          'estimated final time
 ID = scanarray#(4)
                         'specimen ID
 n\% = Readline(handle\%, buf\$, 79)
 n\% = Scan (buf\$, "\%s>\%s[xt44]\%s[xt44]\%4f[x]", STARTDATE\$,
            ,STARTIME$,scanarray#())
 CTIME = scanarray #(0)
                                     'the time
 VL = scanarray#(1)
                                     'the load
 VSA0 = scanarray#(2)
                                     'the initial strain-A
 VSB0 = scanarray#(3)
                                     'the initial strain-B
 n% = Scan(STARTDATE$, "%s>%i[x]%i[x]", STARTMON%, STARTDAY%)
 n% = Scan(STARTIME$, "%s>%i[x]%i[x]%i[x]", STARTHR%
           ,STARTMIN%,STARTSEC%)
 i..dummy% = SetCtrlVal%(hPanel%, CT.Sdate%, STARTDATE$)
 i..dummy% = SetCtrlVal%(hPanel%, CT.Stime%, STARTIME$)
k..err% = ConfirmPopup ("Would you like reload stored file to update plot?")
```

```
k..err% = ConfirmPopup ("Would you like reload stored file to update plot?"

IF k..err% = 1 THEN

Ikeep% = 0

Pts% = 0

WHILE quitloop1% = 0

n\% = Readline (handle%, buf$, 79)
```

```
IF n\% = -2 THEN
    quitloop1\% = 1
  ELSE
  END IF
  n\% = Scan (buf\$, "\%s>\%s[xt44]\%s[xt44]\%5f[x]"
              ,STARTDATE$,STARTIME$,scanarray#())
  CDATA#(1, Ikeep\%) = scanarray#(1)
  CDATA#(3, Ikeep\%) = scanarray#(3)
  CDATA#(4, Ikeep\%) = scanarray#(4)
  IF Ikeep\% < 4 or Ikeep\% > 498 THEN
   Ikeep\% = 4
  ELSE
  ENDIF
  X#(pts\%) = scanarray#(0)
  Y # (pts\%) = scanarray # (3) - VSB0
  Ikeep\% = Ikeep\% + 1
  Pts\% = Pts\% + 1
 WEND
 i..dummy% = PlotXY% (hPanel%,CT.creepcurve1%,X#(),Y#())
                        ,2000,4,4,2,2,1,12)
ELSE
Ikeep\% = 4
END IF
i..dummy% = SetCtrlVal%(hPanel%, CT.Pts%, Pts%)
```

ELSE

i..dummy% = SetActiveCtrl%(CT.Ilength%)
i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "Please input specimen length:")
i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "Specimen area:")
i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "Estimate final strain:")
i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "Specimen ID:")
i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "Estimeat final time:")

k..err% = GetUserEvent% (1, hPanel%, ctrl%) i..dummy% = GetCtrlVal (hPanel%, CT.Ilength%, L0) i..dummy% = GetCtrlVal (hPanel%, CT.Iarea%, A0) i..dummy% = GetCtrlVal (hPanel%, CT.Fstrain%, FS) i..dummy% = GetCtrlVal (hPanel%, CT.Ftime%, FT) i..dummy% = GetCtrlVal (hPanel%, CT.ID%, ID)

```
quitloop1% = 0

i..dummy% = SetCtrlVal%(hPanel%, CT.mw%,

"***ABOUT 1000 DATA TAKEN")

i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "BUT MORE AT FIRST

STAGE***")

CDATA#(0, 0) = L0

CDATA#(1, 0) = A0

CDATA#(1, 0) = FS

CDATA#(2, 0) = FS

CDATA#(3, 0) = FT

CDATA#(4, 0) = ID
```

WHILE quitloop 1% = 0

CTIME = 0

CALL get.data(VL, VSA, VSB, TC1, TC2)

```
i..dummy% = SetCtrlVal%(hPanel%, CT.Ctime%, CTIME)
i..dummy% = SetCtrlVal%(hPanel%, CT.Cstress%, VL)
i..dummy% = SetCtrlVal%(hPanel%, CT.Cstraina%, VSA)
i..dummy% = SetCtrlVal%(hPanel%, CT.Cstrainb%, VSB)
i..dummy% = SetCtrlVal%(hPanel%, CT.Tc1%, TC1)
i..dummy% = SetCtrlVal%(hPanel%, CT.Tc2%, TC2)
i..dummy% = SetCtrlVal%(hPanel%, CT.Sdate%, DATE$)
i..dummy% = SetCtrlVal%(hPanel%, CT.Stime%, TIME$)
```

k..err% = ConfirmPopup ("Is this the starting point?")

IF k..err% = 1 THEN

```
CDATA#(2, 1) = VSA0

CDATA#(3, 1) = VSB0

CDATA#(4, 1) = TC10

CDATA#(5, 1) = TC20

quitloop1\% = 1
```

ELSE

quitloop1% = 0

END IF

WEND

```
handle%=OpenFile (FILENAME$,0, 1, 1)
IF handle\% = -1 THEN
i..dummy% = SetCtrlVal% (hPanel%,CT.mw%,"error opening file")
STOP
ELSE
END IF
FOR I\%=0 TO 4
n\%=FmtFile (handle%, "%s<%f[w0p4]%s",CDATA#(I%,0),dspace$)
NEXT I%
i..dummy% = SetCtrlVal% (hPanel%,CT.mw%,"writing initial data to file")
n\% = Writefile (handle%, CHR$(10), 1)
IF n\% = -1 THEN
 i..dummy% = SetCtrlVal% (hPanel%,CT.mw%,"error writing to file")
ELSE
END IF
n\% = CloseFile (handle%)
```

```
count% = 0
Ikeep% = 1
Pts% = 1
CALL write.data (FILENAME$)
```

```
Ikeep\% = 4
```

END IF

```
quitloop1% = 0
VINC = FS * L0 * 10*1000 / .1 / 300 ' the criteria of taking data
datapoints1% = 700
datapoints2% = 300
StressChange% = 10
```

```
TempChange\% = 20
count\% = 0
                       ' final time / 400
TINC = FT/400
                       ' time indicator of whether it is time to take data
TNEXT = TINC
lavg\% = 0
VAVG = 0
If ull \% = 0
print "tnext="; tnext
i..dummy% = SetCtrlVal% (hPanel%, CT.Ilength%, L0)
i..dummy% = SetCtrlVal% (hPanel%, CT.Iarea%, A0)
i..dummy% = SetCtrlVal% (hPanel%, CT.ID,ID)
i..dummy% = SetCtrlVal% (hPanel%, CT.Fstrain%, FS)
i..dummy% = SetCtrlVal% (hPanel%, CT.Ftime%, FT)
i..dummy% = SetCtrlVal% (hPanel%, CT.mw%, "Press Go for starting test or ")
i..dummy% = SetCtrlVal% (hPanel%, CT.mw%, "Press Quit for stop .....")
```

WHILE quitloop 1% = 0

k..err% = GetUserEvent% (1, hPanel%, ctrl%)

SELECT CASE ctrl%

CASE CT.quit%

quitloop1% = ConfirmPopup% ("Do you want to quit??")

CASE ELSE

WHILE quitloop 1% = 0

k..err% = GetUserEvent% (0, hPanel%, ctrl%)

SELECT CASE ctrl%

CASE CT.quit%

quitloop1% = ConfirmPopup% ("Do you want to quit??")

CASE CT.zoomin%

i..dummy% = SetCtrlVal% (hPanel%, CT.mw%, "Set Mintime and Maxtime") i..dummy% = SetCtrlVal% (hPanel%, CT.mw%, "and Press Zoomin again") n% = SetActiveCtrl (CT.MinTime%)

```
i..dummy% = SetCtrlVal% (hPanel%, CT.mw%, "Press Zoomout to return")
```

i..dummy% = SetCtrlVal% (hPanel%, CT.mw%,"to auto time-scaling mode")

CASE CT.zoomout%

n% = ConfigureAxes (hPanel%,CT.creepcurve1%,1,0,0,1,0,0) i..dummy% = SetCtrlVal% (hPanel%, CT.mw%, "Press Go for continuing")

CASE CT.VarChange%

n% = HidePanel (hPanel%) n% = InstallPopup (varPanel%) quitloop3% = 0

WHILE quitloop3% = 0

k..err% = GetPopupEvent% (1,ctrl%)

SELECT CASE ctrl%

CASE VC.rate%

n% = PromptPopup ("Enter New rate value: ",buf\$,10) n% = Scan (buf\$, "%s>%f[x]", RATE) n% = SetCtrlVal% (varPanel%, VC.newrate%,RATE) n% = SetActiveCtrl% (VC.ppchan%)

CASE VC.ppchan%

n% = PromptPopup ("Enter New PtsPerChan value: ",buf\$,10) n% = Scan (buf\$, "%s>%i[x]", PTSPERCHAN%) n% = SetCtrlVal% (varPanel%, VC.newppc%,PTSPERCHAN%) n% = SetActiveCtrl% (VC.SChange%) NUMPOINTS% = NUMCHANS%*PTSPERCHAN%

CASE VC.SChange%

n% = PromptPopup ("Enter New Stress Changes:(Default value is 10)" ,buf\$,10) n% = Scan (buf\$, "%s>%i[x]", StressChange%) n% = Scan(buf\$, "%s>%i[x]", StressChange%)

n% = SetCtrlVal% (varPanel%, VC.NewSC%, StressChange%)

n% = MessagePopup ("Be sure to change VINC1 before you start !!")

n% = SetActiveCtrl% (VC.TChange%)

CASE VC.TChange%

n% = PromptPopup ("Enter new Temp Changes: (Default value is 20)" ,buf\$,10)

n% = Scan (buf\$, "%s>%i[x]", TempChange%)

n% = SetCtrlVal% (varPanel%, VC.NewTC%, TempChange%)

n% = MessagePopup ("Be sure to change VINC1 before you start !!")

n% = SetActiveCtrl% (VC.VINC1%)

CASE VC.VINC1%

$$\label{eq:n%} \begin{split} &n\% = MessagePopup ("VINC1 = FS * L0 * 10*1000 / .1 / datapoints1") \\ &n\% = PromptPopup ("Enter new VINC1 value: (# of datapoints) ",buf$,10) \\ &n\% = Scan (buf$, "%s>%i[x]", datapoints1%) \\ &VINC1 = FS * L0 * 10*1000 / .1 / datapoints1% \\ &n\% = SetCtrlVal\% (varPanel\%, VC.NewVINC1\%,VINC1) \\ &n\% = SetActiveCtrl\% (VC.OK\%) \end{split}$$

CASE VC.VINC2%

$$\label{eq:n%} \begin{split} &n\% = MessagePopup ("VINC2 = FS * L0 * 10*1000 / .1 / datapoints2") \\ &n\% = PromptPopup ("Enter new VINC2 value: (# of datapoints) ",buf$,10) \\ &n\% = Scan (buf$, "%s>%i[x]", datapoints2%) \\ &VINC2 = FS * L0 * 10*1000 / .1 / datapoints2% \\ &n\% = SetCtrlVal\% (varPanel\%, VC.NewVINC2\%, VINC2) \\ &n\% = SetActiveCtrl\% (VC.OK\%) \end{split}$$

CASE ELSE

quitloop3% = 1

END SELECT

WEND

n% = RemovePopup(0) n% = DisplayPanel (hPanel%) i..dummy% = SetCtrlVal% (hPanel%, CT.rate%, Rate) i..dummy% = SetCtrlVal% (hPanel%, CT.ppc%, PTSPERCHAN%)

CASE CT.storedata%

Ikeep% = Ikeep% + 1 Pts% = Pts% + 1 CDATA#(0, Ikeep%) = CTIME

```
CDATA#(1, Ikeep%) = VL
CDATA#(2, Ikeep%) = VSA
CDATA#(3, Ikeep%) = VSB
CDATA#(4, Ikeep%) = TC1
CDATA#(5, Ikeep%) = TC2
CALL write.data(FILENAME$)
Ikeep% = Ikeep% + count%
count% = 0
```

CASE ELSE

```
i..dummy% = SetCtrlVal%(hPanel%, CT.mw%, "Creep test is proceeding...")
```

```
n\% = Scan (DATE\$, "\%s>\%i[x]\%i[x]", NOWMONTH\%, NOWDAY\%)
n\% = Scan (TIME\$, "\%s>\%i[x]\%i[x]\%i[x]", NOWHR\%, NOWMIN\%,
          NOWSEC%)
SELECT CASE NOWMONTH%
 CASE 1,2,4,6,8,9,11
 NDAYS% = NOWDAY% - STARTDAY% + (NOWMONTH%-
           STARTMON%)*31
 CASE 3
NDAYS% = NOWDAY% - STARTDAY% + (NOWMONTH%-
           STARTMON%)*28
 CASE 5.7.10.12
NDAYS% = NOWDAY% - STARTDAY% + (NOWMONTH%-
           STARTMON%)*30
END SELECT
 CALL get.data(VL, VSA, VSB,TC1, TC2)
 CTIME = (NDAYS% * 24)+(NOWHR%-STARTHR%)+
     (NOWMIN%-STARTMIN%)/60+(NOWSEC%-STARTSEC%)/3600
 IF X#(lkeep%-1)- CTIME = 0 THEN
   recently = 0
 ELSEIF X#(Ikeep%-3)-X#(Ikeep%-1) = 0 THEN
   strainrate = 0
 ELSE
  recently=(Y#(Ikeep%-1)-VSB+VSB0)*0.00001/L0/
      (X#(Ikeep%-1)-CTIME)/3600
  strainrate=(Y#(Ikeep%-3)-Y#(Ikeep%-1))*0.00001/L0/
      (X#(Ikeep\%-3)-X#(Ikeep\%-1))/3600
 END IF
```

```
i..dummy% = SetCtrlVal%(hPanel%, CT.sr%, strainrate)

i..dummy% = SetCtrlVal%(hPanel%, CT.recently%, recently)

i..dummy% = SetCtrlVal%(hPanel%, CT.Ctime%, CTIME)

i..dummy% = SetCtrlVal%(hPanel%, CT.Cstraina%, VSA)

i..dummy% = SetCtrlVal% (hPanel%, CT.Cstrainb%, VSB)

i..dummy% = SetCtrlVal% (hPanel%, CT.Cstrainb%, VSB)

i..dummy% = SetCtrlVal% (hPanel%, CT.Tc1%, TC1)

i..dummy% = SetCtrlVal% (hPanel%, CT.Tc2%, TC2)

i..dummy% = SetCtrlVal% (hPanel%, CT.Pts%, Pts%)

i..dummy% = SetCtrlVal% (hPanel%, CT.count%, count%)

VS = ABS (VSB - CDATA#(3,lkeep%))

SC = ABS (VL - CDATA#(1,lkeep%-3))

TC = ABS (TC1 - CDATA#(4,lkeep%-3))
```

- REM Check if the stress changes greater than StressChanges%
- REM (Default value is 10) OR
- REM the temperature changes greater than TempChange%
- REM (Default value is 20)

IF SC > StressChange% OR TC > TempChange% THEN
VINC1 = FS * L0 * 10*1000 /0.1/ datapoints1%
VINC = VINC1
ELSE
VINC2 = FS * L0 * 10*1000 / .1 / datapoints2%
VINC = VINC2
END IF
i..dummy% = SetCtrlVal% (hPanel%, CT.VINC%, VINC)

IF VS > VINC AND VINC = VINC1 THEN

```
Ikeep% = Ikeep% + 1

Pts% = Pts% + 1

count% = count% + 1

CDATA#(0, Ikeep%) = CTIME

CDATA#(1, Ikeep%) = VL

CDATA#(2, Ikeep%) = VSA

CDATA#(3, Ikeep%) = VSB

CDATA#(4, Ikeep%) = TC1

CDATA#(5, Ikeep%) = TC2
```

```
ELSEIF VS > VINC AND VINC = VINC2 THEN
CALL average.data (Iavg%,VAVG,CTIME,VL,VSA,VSB,TC1)
i..dummy% = SetCtrlVal% (hPanel%,CT.Iavg%,Iavg%)
```

IF ABS (VAVG - CDATA#(3,Ikeep%)) > VINC AND ABS(VAVG) > 0

THEN

```
Iavg\% = 0
   If ull \% = 0
   Ikeep\% = Ikeep\% + 1
   Pts\% = Pts\% + 1
   CDATA#(0, Ikeep\%) = CTIME
   CDATA#(1, Ikeep\%) = VL
   CDATA#(2, Ikeep\%) = VSA
   CDATA#(3, Ikeep%) = VSB
   CDATA#(4, Ikeep\%) = TC1
   CDATA#(5, Ikeep\%) = TC2
   CALL write.data (FILENAME$)
   Ikeep\% = Ikeep\% + count\%
   count\% = 0
  ELSE
  END IF
ELSE
END IF
```

```
TNEXT = CTIME+ TINC
i..dummy% = SetCtrlVal% (hPanel%,CT.Iavg%,Iavg%)
```

```
IF Ikeep% > 498 THEN
CALL write.data (FILENAME$)
count% = 0
Ikeep% = 3
ELSE
END IF
```

```
END SELECT
WEND
```

END SELECT WEND

```
n%=CloseFile (handle%)
k..err% = Init.DA.Brds% (1, boardtype%)
k..err% = CloseInterfaceManager%
```

```
SUB get.data (VL, VSA, VSB, TC1, TC2)
```

```
k..err% = SCAN.Op% (1.NUMCHANS%, chans%(), gains%(),
                     data.buf%(),NUMPOINTS%,RATE,0.0#)
k..err% = DAQ.Scale (1,1,numpoints%,data.buf%(),volts.buf#())
J\% = 0
VL = 0
VSA = 0
VSB = 0
TC1 = 0
TC2 = 0
WHILE J% < NUMPOINTS% -1
  VL = VL + volts.buf#(J\%)
  VSA = VSA + volts.buf#(J\% + 1)
  VSB = VSB + volts.buf#(J\% + 2)
  TC1 = TC1 + volts.buf#(J\% + 3)
  TC2 = TC2 + volts.buf#(J\% + 4)
  J\% = J\% + NUMCHANS\%
WEND
J% = NUMPOINTS%/NUMCHANS%
VL = (VL/J\%)*1000 - 5
VSA = (VSA/J\%)*1000
VSB = (VSB/J\%)*1000
TC1 = (TC1/J\%)*1000
TC2 = (TC2/J\%)*1000*100
```

```
END SUB
```

```
SUB write.data (FILENAME$)
handle%=OpenFile (FILENAME$,0, 1, 1)
IF handle% = -1 THEN
i..dummy% = SetCtrlVal% (hPanel%,CT.mw%,"error opening file")
STOP
ELSE
END IF
Ikeep% = Ikeep% - count%
FOR K% = 0 TO count%
n%=FmtFile (handle%, "%s<%s[w14]%s[w2]%s[w9]%s[w2]"
,DATE$,dspace$,TIME$,dspace$)
FOR I%=0 TO 4
n%=FmtFile (handle%, "%s<%f[w9p2]%s[w2]"</pre>
```

,CDATA#(I%,Ikeep%+K%),dspace\$) NEXT I% i..dummy% = SetCtrlVal% (hPanel%,CT.mw% ,"writing data to file") i..dummy% = SetCtrlVal% (hPanel%,CT.count%,K%) n%=Writefile (handle% , CHR\$(10),1) IF n% = -1 THEN i..dummy% = SetCtrlVal% (hPanel%,CT.mw% ,"error writing to file") ELSE END IF X#(Ikeep%+K%) = CDATA#(0, Ikeep%+K%) - VSB0

i..dummy% = PlotXY% (hPanel%,CT.creepcurve1%,X#(),Y#(),2000,4,4,2,2,1,12)

NEXT K% n% = CloseFile(handle%)

END SUB

SUB average.data (Iavg%,VAVG,CTIME,VL,VSA,VSB,TC1)

```
AVG\#(0, Iavg\%) = CTIME
AVG#(1, Iavg\%) = VL
AVG#(2, Iavg\%) = VSA
AVG#(3, Iavg\%) = VSB
AVG#(4, Iavg\%) = TC1
Iavg\% = Iavg\% + 1
IF Iavg\% = 20 THEN
 VL = 0
 VSA = 0
 VSB = 0
 TC1 = 0
 FOR I\% = 0 TO 19
   VL = VL + AVG\#(1, 1\%)
   VSA = VSA + AVG\#(2, I\%)
   VSB = VSB + AVG\#(3, I\%)
   TC1 = TC1 + AVG#(4, I\%)
 NEXT I%
 AVG#(0,20) = AVG#(0,0)
 CTIME = (AVG\#(0, Iavg\%-1) + AVG\#(0, Iavg\%))/2
 VL = VL/20
 VSA = VSA/20
```

```
VSB = VSB/20
        VAVG = VSB
        TC1 = TC1/20
        If ull\% = 1
        Iavg\% = 0
      ELSEIF Ifull% = 1 THEN
           VL = 0
           VSA = 0
           VSB = 0
           TC1 = 0
           FOR I% = 0 TO 19
             VL = VL + AVG\#(1, I\%)
             VSA = VSA + AVG\#(2, I\%)
             VSB = VSB + AVG\#(3, I\%)
             TC1 = TC1 + AVG\#(4, I\%)
           NEXT I%
           AVG#(0,20) = AVG#(0,0)
           CTIME = (AVG\#(0,Iavg\%-1)+AVG\#(0,Iavg\%))/2
           VL = VL/20
           VSA = VSA/20
           VSB = VSB/20
           VAVG = VSB
           TC1 = TC1/20
      ELSE
        VAVG = 0
END IF
```

END SUB

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