AN INVESTIGATION OF THE LOW TEMPERATURE CREEP DEFORMATION BEHAVIOR OF TITANIUM GRADE 7 AND GRADE 5 ALLOYS—PROGRESS REPORT WITH PRELIMINARY RESULTS

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ABSTRACT

The U.S. Nuclear Regulatory Commission (NRC) is interacting with U.S. Department of Energy (DOE) to gain insight into the potential geologic repository at Yucca Mountain, Nevada. In the design DOE is currently considering, the radioactive materials would be encapsulated in waste packages and emplaced in drifts (tunnels) below the surface. Subsequently, drip shields, primarily made of Titanium Grade 7 and Grade 24, would be placed over the waste packages to protect them from seepage and rockfall. If drift degradation occurs, drip shields would be subjected to impact loads due to the falling rock and static and dynamic (seismic) loads resulting from accumulated rockfall rubble. In the case when the accumulated rockfall rubble does not result in static loads high enough to produce immediate failure of the drip shield, the drip shield components would be subjected to permanent static loading, leading to stresses that may approach or exceed the yield stress of the drip shield materials. Titanium Grade 7 and Grade 5 (surrogate of Titanium Grade 24) were investigated for low temperature {from 25 to 250 °C [77 to 482 °F]} tensile and creep behavior. The tensile behavior of Titanium Grade 7 exhibited flow stress drops that increased with an increase in temperature. This type of tensile behavior was attributed to a rise in the mobile dislocation density. The tensile behavior of Titanium Grade 5 exhibited work hardening at 250 °C [482 °F] and no work hardening at lower temperatures. This type of tensile behavior was attributed to a convergence of the critical stress for slip on various slip systems. Moreover, for Titanium Grade 7, there was no change in the amount of creep strain with an increase in temperature at 85 percent of the respective yield stress. This type of creep behavior was attributed to a change in the deformation mechanisms from slip and twinning at lower temperatures {25 to 50 °C [77 to 122 °F]} to solely slip at higher temperatures {100 to 150 °C [212 to 302 °F]}. Results of the experimental tests indicated that creep initiation for Titanium Grade 7 and Grade 5 occurs for stresses below the yield stress of a corresponding material.

CONTENTS

CONTENTS (continued)

FIGURES

FIGURES (continued)

FIGURES (continued)

TABLES

ACKNOWLEDGMENTS

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QUALITY OF THE DATA, ANALYSES, AND CODE DEVELOPMENT

DATA: All CNWRA-generated data contained in this report meet quality assurance requirements described in the Geosciences and Engineering Division Quality Assurance Manual. Sources of other data should be consulted for determining the level of quality of those data. The work presented in this report is documented in Scientific Notebook 893. The experimental tests on creep and tensile behavior of Titanium Grade 7 and Grade 5 were performed at Westmoreland Mechanical Testing and Research, Inc. The chemical analysis of Titanium Grade 7 and Grade 5 was conducted by Staveley Services Materials Testing. Both subcontractors have a quality assurance program accepted by CNWRA.

ANALYSES AND CODES: Spreadsheet calculations were performed using Microsoft® Office Excel® 2003 (Microsoft Corporation, 2003).

REFERENCE:

Microsoft Corporation. “Microsoft® Office Excel® 2003.” Redmond, Washington: Microsoft Corporation. 2003.

EXECUTIVE SUMMARY

The drip shield, primarily made of Titanium Grade 7 and Grade 24, is part of several engineering barrier subsystems that the U.S. Department of Energy (DOE) is considering for the potential geologic repository at Yucca Mountain, Nevada, where the radioactive waste materials would be encapsulated in waste packages and emplaced in drifts (tunnels) below the surface. The main function of the drip shields is to protect the waste packages from seepage and rockfall. If drift degradation would occur, drip shields would be subjected to impact loads due to the falling rock and static and dynamic (seismic) loads resulting from accumulated rockfall rubble. When the accumulated rockfall rubble does not result in static loads high enough to produce immediate failure of the drip shield, the drip shield components would be subjected to permanent loads that might cause stresses approaching or exceeding the yield stress of the drip shield materials.

A literature review previously performed at the Center for Nuclear Waste Regulatory Analyses indicated that surrogates of Titanium Grade 7 and Grade 24 exhibit creep failure when subjected to stresses close to the yield stress for an extended period of time (Ankem and Wilt, 2006). Moreover, the review indicated that there is a lack of specific information on creep behavior of these alloys to draw definite conclusions. Therefore, a study was performed to investigate the creep deformation behavior of titanium alloys, currently considered by DOE as drip shield materials, at temperatures ranging from 25 to 250 °C [77 to 482 °F] and at stress levels of 40 to 100 percent yield stress. Because Titanium Grade 24 is not readily available, Titanium Grade 5 (Ti-6Al-4V) was used as a surrogate. The basis for using Titanium Grade 5 is that the mechanical properties of both alloys are very similar, although the chemical composition of Titanium Grade 24 includes a small amount of palladium to enhance corrosion resistance properties.

This study included the evaluation of the creep behavior of the materials as well as the determination of the activation energy necessary for creep deformation, the characterization of the as-received plate microstructures using optical and scanning electron microscopy, and the transmission electron microscopy of the creep specimens. In particular, the creep testing was performed at various stress levels—40, 55, 70, 85, and 100 percent yield stress at 150 °C [302 °F].[1] At this temperature, the empirical equation that best describes the creep behavior were determined. Also at this temperature, the threshold stress for creep, or the stress below which creep is considered to be negligible was calculated. Further, creep testing was performed at various temperatures {25 °C [77 °F], 50 °C [122 °F] (for Titanium Grade 7 only), 100 °C [212 °F], 150 °C [302 °F], and 250 °C [482 °F] (for Titanium Grade 5 only)} at a constant stress level of 85 percent yield stress. Using this creep data, the activation energy for creep was calculated. Because the mechanical properties of titanium alloys are highly dependent on the microstructure (i.e., grain size and morphology), as-received plate materials were examined by optical and scanning electron microscopy to determine the grain size of Titanium Grade 7 and Grade 5 and whether the materials have any rolling texture. The transmission electron microscopy investigation of the undeformed and creep-deformed material was performed to determine the crystal structure of the alloys and the creep deformation mechanisms.

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[1] DOE has used material properties associated with a temperature of 150 °C [302 °F] for structural performance assessment of drip shield systems. (Bechtel SAIC Company, LLC, 2004)

The analysis of creep tests performed for Titanium Grade 7 and Grade 5 at various stress levels at 150 °C [302 °F] shows that, for both alloys, the creep behavior is best described by a logarithmic equation of the type $\varepsilon = A' + B\ln(t)$, where ε is creep strain that depends on time t, and A' and B are constants. Moreover, it was found that the creep constants A' and B increase exponentially with an increase in stress level. Furthermore, it was determined that specimens of Titanium Grade 5 exhibit much less creep than Titanium Grade 7. While the Titanium Grade 5 specimen tested at 100 percent yield stress crept to approximately 1.3 percent, the specimens tested at lower stress levels crept to less than 0.1 percent. As such, while the data can be described by logarithmic type empirical equations, the fit obtained for Titanium Grade 5 does not describe data as well as the one obtained for Titanium Grade 7. The creep strain was plotted as a function of stress level, and it was found that creep strain decreases exponentially as stress level decreases. The threshold stresses for creep of Titanium Grade 7 and Grade 5 at 150 °C [302 °F] were found to be approximately 32.3 and 44.9 percent yield stress, respectively.

Preliminary test results showed unexpected creep behavior for Titanium Grade 7 when tested at 85 percent of the respective yield stress at various temperatures. It was found that, after the early stages of creep, the creep strain rate decreases as the temperature increases above 50 °C [122 °F]. This behavior is unexpected because creep strain rate usually increases with temperature when the creep deformation mechanism is thermally activated, such as for slip. The increase in creep strain rate with decreasing temperature is thus attributed to increased twinning activity at 25 to 50 °C [77 to 122 °F] compared to the higher temperatures. The load drop after yielding at higher temperatures could be another contributing factor to this behavior. The calculated activation energy for creep of Titanium Grade 7 at the early stage of creep is consistent with previously measured values for slip-controlled creep in α-titanium (Tung and Sommer, 1970; Zeyfang, et al., 1971), and the slight increase in activation energy with increasing strain suggests that at 25 to 50 °C [77 to 122 °F] twinning is a creep deformation mechanism at the later stages of creep.

Plates of as-received Titanium Grade 7 and Grade 5 were polished and etched on the various faces to reveal the grain structure. Prepared specimens were examined using optical microscopy for Titanium Grade 7 and scanning electron microscopy for Titanium Grade 5. For Titanium Grade 7, the grains were equiaxed (have equal axial length) and there was no elongation in the rolling direction; for Titanium Grade 5, the heat treatment of the alloy produced a mill-annealed microstructure of alternating layers (bands) of equiaxed α grains with transformed β grains and elongated α grains with intergranular β grains.

Undeformed, as-received Titanium Grade 7 specimens were examined, confirming that this alloy has a small number of dislocations in as-received specimens. In addition, creep deformed specimens tested at stress levels of 100, 85, and 70 percent yield stress at 150 °C [302 °F] were examined. The specimen tested at 100 percent yield stress crept significantly to a strain of approximately 13.7 percent; *a*-type prism slip and twinning were found to be active deformation mechanisms in this specimen. For the specimens tested at 150 °C [302 °F] at the stress levels of 85 and 70 percent yield stress, *a*-type prism slip was the only observed deformation mechanism, and no twinning was identified. This is likely because of the limited extent of strain at the lower stress levels. Also the specimens tested at 25 , 50, and 100 °C [77, 122, and 212 °F] at 85 percent yield stress were examined in this investigation. For the specimens examined at 25 and 50 °C [77 and 122 °F], *a*-type prism slip and twinning were found to be active deformation mechanisms, whereas the specimen tested at 100 °C [212 °F] deformed only by *a*-type slip.

Undeformed, as-received Titanium Grade 5 specimens were examined, confirming that the α and β phases have a Burgers orientation relationship to one another. The mechanical behavior of the creep deformed specimens is controlled by the α-phase due to the high volume fraction of this phase in Titanium Grade 5. At all temperatures and stress levels, *a*-type slip was identified as the predominant deformation mechanism in the α-phase. At temperatures below 250 °C [482 °F], the dislocation morphology was found to be a coarse planar slip. In the specimen tested at 250 °C [482 °F], the slip was less planar and random dislocation tangles (places where dislocations have become wrapped and knotted around one another) were identified. Further, in all specimens except one tested at 100 percent yield stress at 150 °C [302 °F], no deformation products were identified in the β-phase. The limited strain in these specimens (less than 0.1 percent) causes the strain to be accommodated elastically in the β-phase. For the specimen tested at 100 percent yield stress at 150 °C [302 °F], the strain was sufficient that dislocations were found in the β-phase, which has little effect on the overall creep behavior of the alloy.

This investigation showed that it is critical to consider the microstructure and deformation mechanisms, as observed by optical and transmission electron microscopy, when interpreting the data from the creep tests at various temperatures and stress levels to understand the creep behavior of the titanium alloys. Results of the preliminary experimental tests indicated that creep initiation for Titanium Grade 7 and Titanium Grade 5 occurs for stresses below the yield stress of a corresponding material for all evaluated temperatures, however, the conclusions presented in this report are preliminary. Additional experimental tests on creep behavior of Titanium Grade 7 and Grade 5 have been initiated to confirm the findings of this study. More explicit conclusions will be offered in future reports.

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1 INTRODUCTION

1.1 Background

The U.S. Department of Energy (DOE) is studying the Yucca Mountain site in Nevada to determine whether it is suitable for building a geologic repository for the disposal of the nation's spent nuclear fuel and high-level radioactive waste (DOE, 1998). According to the design DOE is currently considering, the radioactive materials would be encapsulated in waste packages and emplaced in drifts (tunnels) below the surface. Subsequently, drip shields primarily made of Titanium Grade 7 and Grade 24 would be placed over the waste packages to protect them from seepage and rockfall. If drift degradation occurs, drip shields would be subjected to impact loads due to the falling rock and static and dynamic (seismic) loads resulting from accumulated rockfall rubble. When the accumulated rockfall rubble does not result in static loads high enough to produce immediate drip shield structural instability, the drip shield components would be subjected to permanent loads that might cause stresses approaching or exceeding the yield stress of the drip shield materials.

DOE indicates that between 200 °C and 315 °C [392 °F and 599 °F], the deformation of many titanium alloys loaded to yield point does not increase with time (Bechtel SAIC Company, LLC, 2004a). Because the expected temperature in the drip shield is less that 300 °C [572 °F], and given that creep rates decrease at lower temperatures, DOE concludes, that creep deformation of the engineered barriers will not occur to any appreciable extent under repository exposure conditions. Therefore, the creep processes in the engineered barriers are excluded by DOE based on a low probability screening criterion. On the contrary, the literature review previously performed by the Center for Nuclear Waste Regulatory Analyses indicated that, for stress levels close to the yield stress, titanium alloys may experience low temperature (less than 25 percent of the melting temperature of the material) creep after several thousands of years (Ankem and Wilt, 2006). In fact, titanium alloys are known to deform slowly over time, or creep, at low temperatures at stresses less than the yield stress of the material (Adenstedt, 1949). This type of behavior may compromise the integrity of the drip shield, leading to a complete loss of its intended functions. As an outcome, early failure of a drip shield system may result in an increase in the amount of water contacting the waste package. Consequently, this may lead to elevated corrosion rates of the waste package and, potentially, to an increase in the rate of radionuclide mobilization and release. Therefore, it is important to gain insight into the creep behavior of titanium alloys DOE is currently considering for the drip shield materials. Prior to this investigation, there has been no systematic study of the low-temperature creep behavior of Titanium Grade 7 or Grade 24 in the range of temperatures (from 25 to 250 °C [77 to 482 °F]) and stress levels (from 40 to 100 percent yield stress) that are of interest for the drip shield performance assessment (Ankem and Wilt, 2006).

1.2 Objectives and Scope

This study focuses on the creep deformation behavior of titanium alloys that DOE is currently considering for drip shield material at temperatures ranging from 25 to 250 °C [77 to 482 °F] and at stress levels ranging from 40 to 100 percent yield stress. Because Titanium Grade 24 is not readily available, Titanium Grade 5 (Ti-6Al-4V) is used as a surrogate, given that the mechanical properties of both alloys are very similar, although the chemical composition of

Titanium Grade 24 includes a small amount of palladium (0.04 to 0.08 weight percent) to enhance corrosion resistance properties.

The creep behavior of both titanium alloys, Grade 7 and Grade 5, is evaluated for a range of stress levels at the constant temperature of 150 °C [302 °F][1] and for a range of temperatures 25 to 250 °C [77 to 482 °F] at the constant stress level of 85 percent yield stress of the corresponding material. This investigation includes (i) analysis of the creep response including determination of the activation energy for creep deformation, (ii) characterization of the as-received plate microstructures using optical and scanning electron microscopy, and (iii) transmission electron microscopy of the creep specimens.

1.3 Outline of the Report

This report is divided into six chapters, with Chapter 1 being the introduction. Chapter 2 describes the investigation of the low temperature creep of Titanium Grade 7 and is further divided into three sections. The first section discusses the analysis of the creep data obtained from specimens tested at the range of temperatures and stress levels. Empirical equations are given to describe the creep data, the threshold stress level for the onset of creep at 150 °C [302 °F] is determined, and the activation energy for creep at 85 percent yield stress is calculated. The second section includes a characterization of the microstructure of the as-received Titanium Grade 7 plate. The results of a transmission electron microscopy investigation of undeformed and creep-deformed Titanium Grade 7 specimens are presented in the third section. Chapter 3 is similar to Chapter 2 but covers Titanium Grade 5. The summary and conclusions are given in Chapter 4. Chapter 5 provides suggestions for future work, and the references are given in Chapter 6.

Three appendices are included. Appendix A presents the results for tensile testing of the Titanium Grade 7 and Grade 5 specimens. Appendix B describes the crystallography and texture of the as-received Titanium Grade 7 and Grade 5 plates. Appendix C presents experimental data of tensile and creep tests performed at Westmoreland Mechanical Testing and Research, Inc. for Titanium Grade 7 and Grade 5.

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[1] DOE has used material properties associated with a temperature of 150 °C [302 °F] for structural performance assessment of drip shield systems. (Bechtel SAIC Company, LLC, 2004b)

2 TITANIUM GRADE 7

Titanium Grade 7 is a commercially pure titanium alloy with an addition of a small amount of palladium to enhance corrosion resistance properties. The material used in this investigation was obtained from Tricor Industrial, Inc., and the chemical analysis by Staveley Services Materials Testing showed that its chemical composition meets the ASTM Standards for Titanium Grade 7 (ASTM B265-06b). Table 2-1 shows the chemical composition as given by Staveley Services Materials Testing. For Titanium Grade 7, the three main tasks in this investigation involve analysis of creep test data; characterization of the microstructure and texture of the undeformed, as-received plate material; and transmission electron microscopy to identify the creep deformation mechanisms.

2.1 Analysis of Creep Data of Titanium Grade 7

To determine the creep behavior of Titanium Grade 7 under various loading conditions, creep tests were performed at Westmoreland Mechanical Testing and Research, Inc. At 150 °C [302 °F], Titanium Grade 7 specimens were creep tested at various stress levels (given as a percentage of the 0.2 percent yield stress; see Appendix A)—40, 55, 70, 85, and 100 percent yield stress. The duration of each test was 200 hours. Moreover, additional Titanium Grade 7 specimens were creep tested at 85 percent of the respective yield stress at various temperatures—25 , 50, 100, and 150 °C [77, 100, 122, and 302 °F]. The objectives of this analysis were to determine (i) the empirical equations that best describe the creep behavior of Titanium Grade 7 at the various stress levels for 150 °C [302 °F], (ii) the creep constants as a function of stress level at 150 °C [302 °F], (iii) minimum stress levels at which creep occurs for 150 °C [302 °F], and (iv) the activation energy for creep of Titanium Grade 7 at 85 percent yield stress.

2.1.1 Determining the Empirical Equation to Describe Creep Data

As customary in materials science, creep behavior of a material can be described by one of two equations of a creep curve (Aiyangar, et al., 2005). The first equation is a power-law equation where creep strain, ε, is related to the time of testing, t, by an equation of the type

$$\varepsilon = At^n \tag{2-1}$$

where A is the creep constant of proportionality, and n is the creep exponent. Alternatively, a creep curve may follow a logarithmic equation of the type:

$$\varepsilon = A' + B\ln(t) \tag{2-2}$$

where A' and B are constants.

In this study, the creep curves were derived using both the power-law Eq. (2-1) and the logarithmic form Eq. (2-2) to determine which empirical equation describes the data more accurately. The accuracy with which the equation describes the creep behavior of a material is measured by the R^2 value (where R is the Pearson product moment correlation coefficient

Table 2-1. Chemical Composition of Titanium Grade 7 Used in This Investigation						
Carbon	**Hydrogen**	**Oxygen**	**Iron**	**Palladium**	**Nitrogen**	**Titanium**
0.01 weight percent	0.001 weight percent	0.13 weight percent	0.08 weight percent	0.16 weight percent	0.01 weight percent	Balance

through the data points). An R^2 value of 1 indicates that the derived equation describes the data ideally, whereas an R^2 value of significantly less than 1, or 0, indicates that the derived empirical equation poorly, or inappropriately correlates the data.

The 200-hour creep curves for Titanium Grade 7 at 150 °C [302 °F] at various stress levels are shown in Figures 2-1 through 2-5, superimposed with the power-law and logarithmic-type equations that describe the experimental data. Figure 2-6 shows a composite creep curve for the specimens tested at the various stress levels.

The creep curves shown in Figures 2-1 through 2-5 indicate that at the lower stress levels (40 and 55 percent yield stress), the power-law Eq. (2-1) described the creep data slightly better than the logarithmic Eq. (2-2). However, once the extent of creep strain becomes more significant at higher stress levels (70, 85, and 100 percent yield stress), the logarithmic equation is more accurate. Therefore, the logarithmic equation will be used to describe the creep behavior for Titanium Grade 7 at 150 °C [302 °F].

2.1.2 Creep Constants as a Function of Stress Level

Given that the creep behavior for Titanium Grade 7 at 150 °C [302 °F] is best described by the logarithmic Eq. (2-2), the creep constants A' and B can be given as a function of stress level. The values for creep constants A' and B, taken from the fitting empirical equations for the creep curves presented in Figures 2-1 through 2-5, are shown in Figures 2-7 and 2-8 as a function of stress level.

Both creep constants, A' and B, can be related to the stress level, σ (where σ is the percentage of the 0.2 percent yield stress), by an exponential equation, as follows

$$A' = 6.90 \times 10^{-4}\, e^{0.0771\sigma} \tag{2-3}$$

$$B = 4.94 \times 10^{-5}\, e^{0.1041\sigma} \tag{2-4}$$

The R^2 value of the Pearson product moment correlation coefficient for A' fitting curve was estimated at 0.80 and for B at 0.9688. This suggests that the B creep constant can be better described by the exponential equation than A' the creep constant. The creep constant A' largely reflects a few measurements made in the first hour of the creep test, which may have great variability. Creep constant B reflects the large number of measurements made after the first hour, which are more consistent.

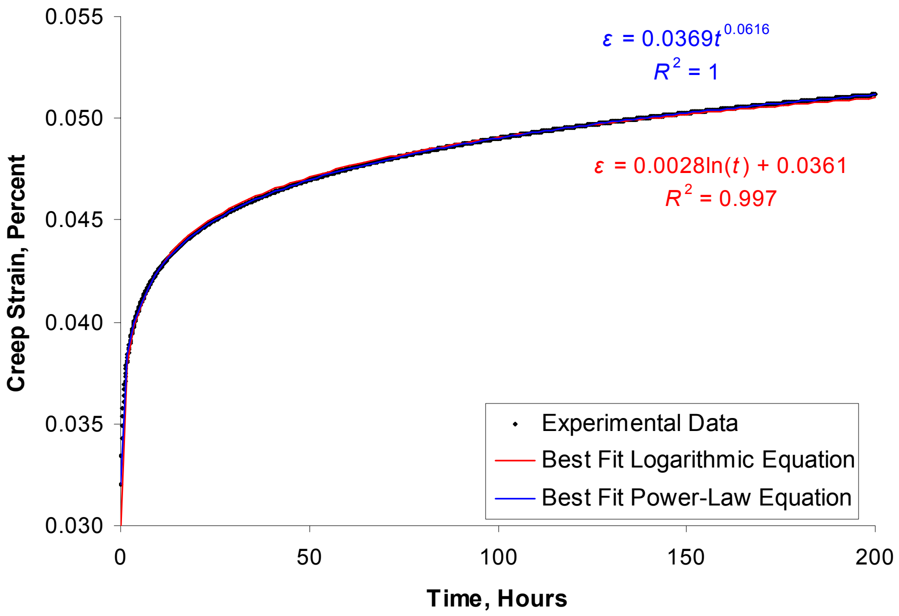


Figure 2-1. Experimental and Analytical Creep Curves for Titanium Grade 7. Stress Level of 40 Percent Yield Stress; Temperature of 150 °C [302 °F].

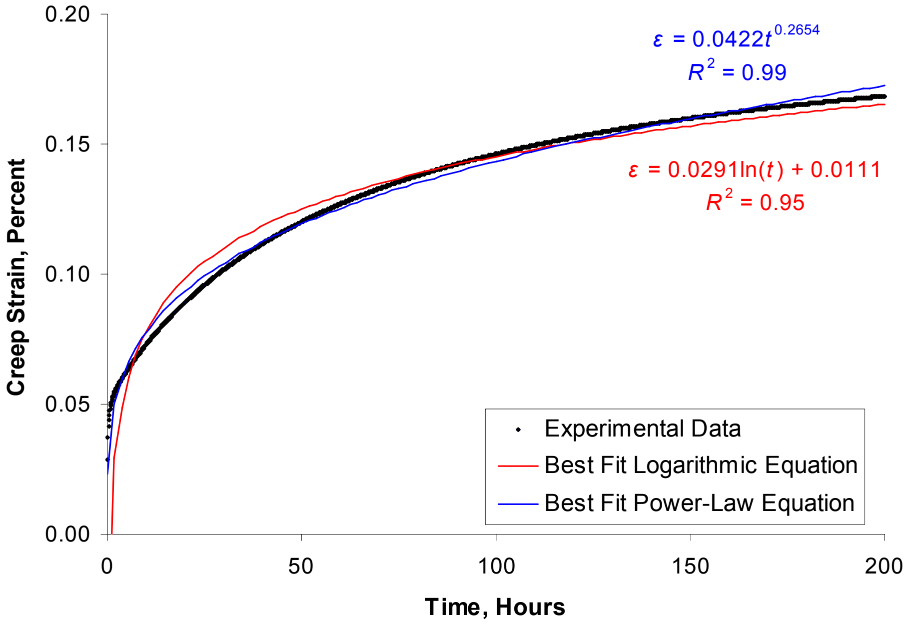


Figure 2-2. Experimental and Analytical Creep Curves for Titanium Grade 7. Stress Level of 55 Percent Yield Stress; Temperature of 150 °C [302 °F].

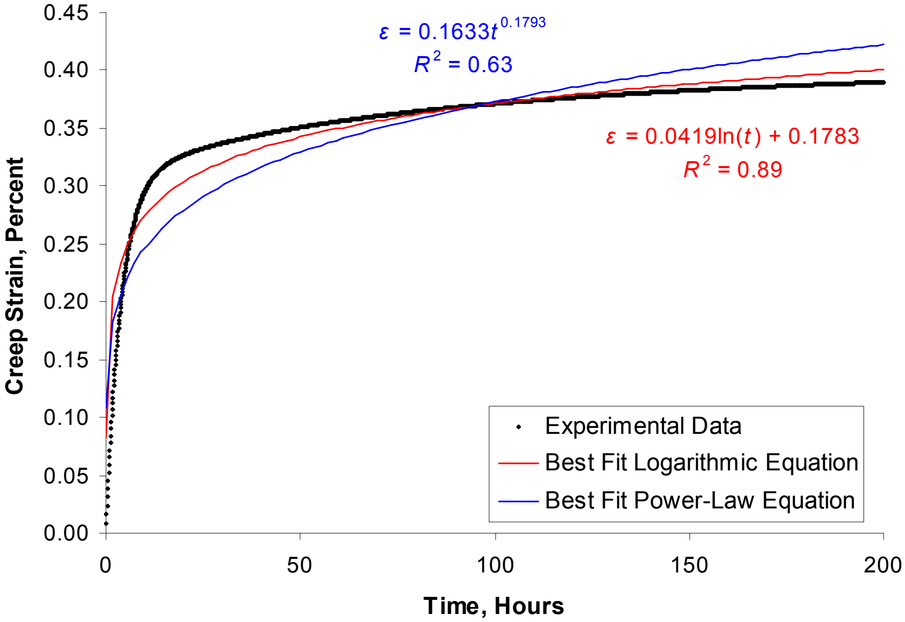


Figure 2-3. Experimental and Analytical Creep Curves for Titanium Grade 7. Stress Level of 70 Percent Yield Stress; Temperature of 150 °C [302 °F].

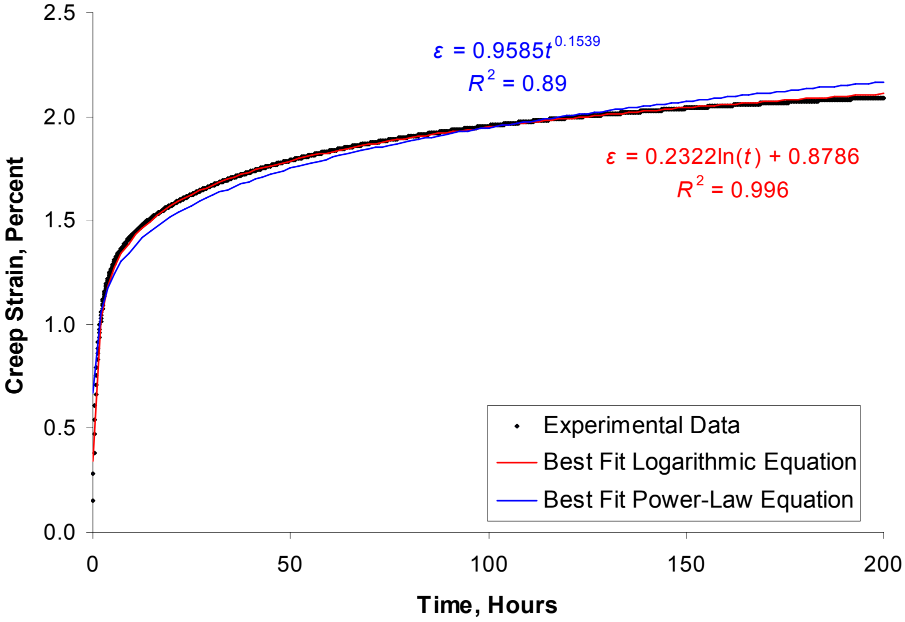


Figure 2-4. Experimental and Analytical Creep Curves for Titanium Grade 7. Stress Level of 85 Percent Yield Stress; Temperature of 150 °C [302 °F].

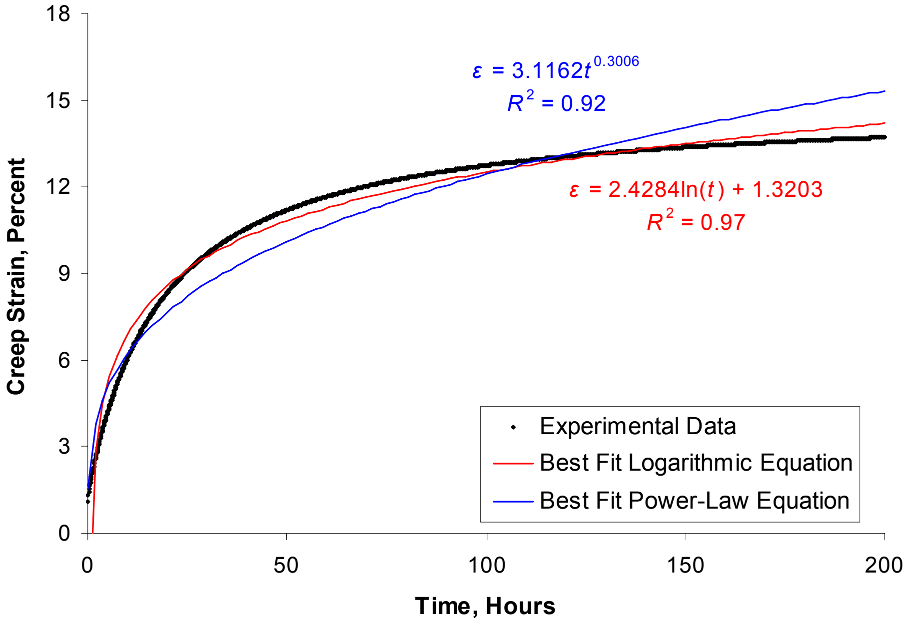


Figure 2-5. Experimental and Analytical Creep Curves for Titanium Grade 7. Stress Level of 100 Percent Yield Stress; Temperature of 150 °C [302 °F].

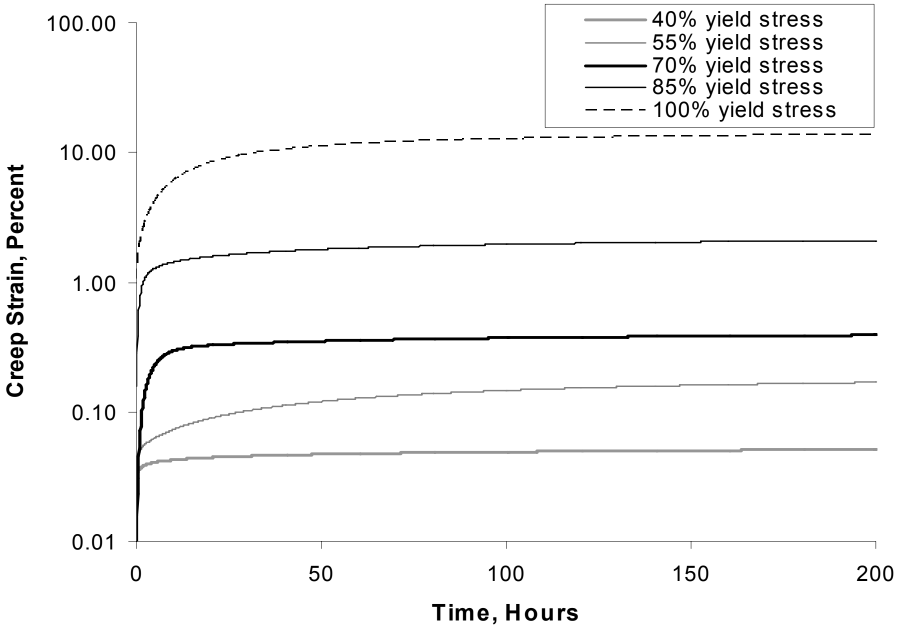


Figure 2-6. Experimental Creep Curves for Titanium Grade 7 at 150 °C [302 °F]

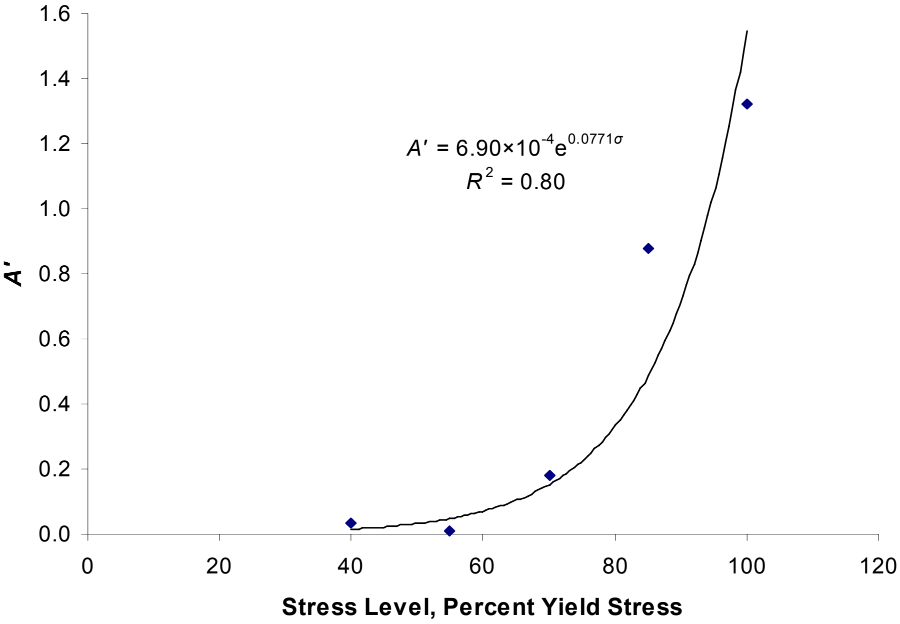


Figure 2-7. The Creep Constant A' as a Function of Stress Level for Titanium Grade 7 Tested at 150 °C [302 °F]

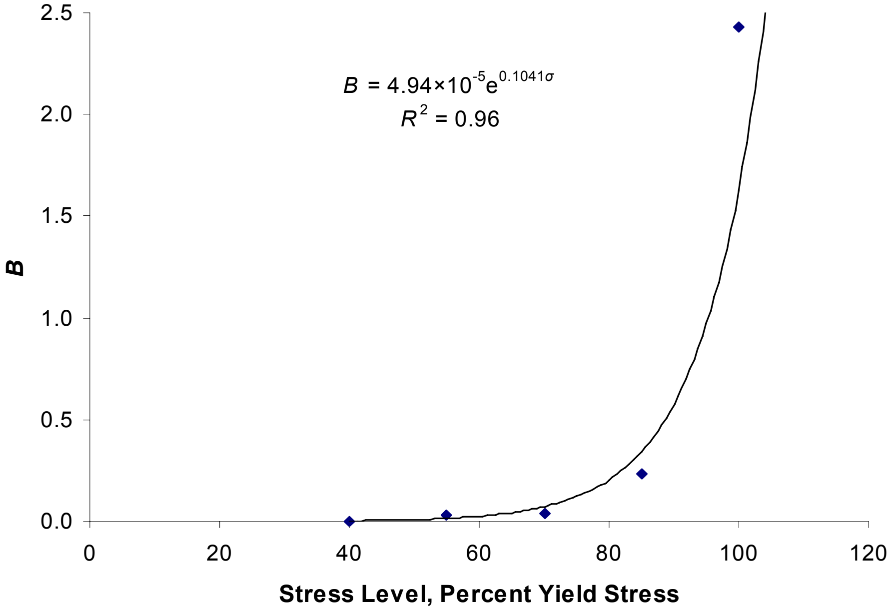


Figure 2-8. The Creep Constant B as a Function of Stress Level for Titanium Grade 7 Tested at 150 °C [302 °F]

Substituting Eqs. (2-3) and (2-4) into Eq. (2-2) gives a general expression for the effect of stress level on creep strain for Titanium Grade 7 at 150 °C [302 °F], as follows

$$\varepsilon = 6.90 \times 10^{-4}\, e^{0.0771\sigma} + 4.94 \times 10^{-5}\, e^{0.1041\sigma} \ln(t) \tag{2-5}$$

2.1.3 Threshold Stress for Creep Deformation of Titanium Grade 7

For commercially pure titanium, Adenstedt (1949) reported creep at a stress level of 60 percent yield stress. However, no definitive creep threshold stress has been reported for Titanium Grade 7. The experimental data from the creep tests performed at Westmoreland Mechanical Testing and Research, Inc. can be used to derive a minimum threshold below which creep deformation is negligible for Titanium Grade 7. It was determined that even at the lowest stress level at which creep testing was performed (40 percent yield stress), Titanium Grade 7 exhibited a creep behavior. Therefore, an empirical equation must be developed to describe the extent of creep strain as a function of stress level. As seen in Figure 2-9, an exponential form [Eq. (2-6)] can be used to describe the relationship between strain after 200 hours, ε, and stress level, σ (where σ is the percentage of the 0.2 percent yield stress)

$$\varepsilon = 1.05 \times 10^{-3}\, e^{0.0913\sigma} \tag{2-6}$$

However, this type of an exponential equation will never produce zero. Therefore, the stress threshold must be redefined as the stress level below which creep strain is considered to be undetectable due to the accuracy of the instruments. From the specifications supplied by Westmoreland Mechanical Testing and Research, Inc., the accuracy of the strain gauge is 0.00508 mm [±0.00020 in]. With a 2.54-cm [1-in] gage length, this corresponds to the accuracy of 0.02 percent. As such, the stress level that corresponds to 0.02 percent strain will be assumed as the stress threshold for creep deformation. Solving Eq. (2-6) for ε = 0.02 percent gives a value for the stress threshold as 32.3 percent yield stress. Therefore, this stress level should be viewed as the stress level below which creep strain in Titanium Grade 7 is negligible.

2.1.4 Activation Energy for Creep Deformation

The activation energy for creep deformation of a material helps to determine the rate-controlling creep deformation mechanism (Miller, et al., 1987). Miller, et al. (1987) have shown that for titanium alloys, the activation energy may change if the deformation mechanism responsible for creep changes. For instance, the creep mechanism may change from slip at low temperatures to self-diffusion at high temperatures.

The activation energy for non-steady-state creep at a constant stress for titanium alloys was first determined by Thompson and Odegard (1973) using Eq. (2-1). They utilized the time derivative of Eq. (2-1) to eliminate the time dependence of the strain rate in order to obtain an empirical equation for strain rate as a function of strain

$$\dot{\varepsilon} = nA^{1/n}(e)^{1-1/n} \tag{2-7}$$

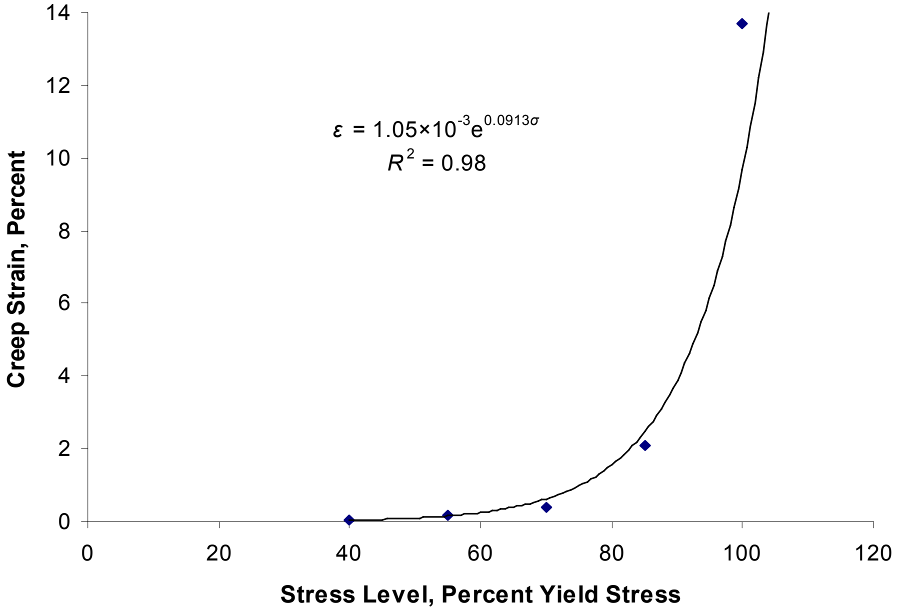


Figure 2-9. A Creep Strain as a Function of Stress Level for Titanium Grade 7 Tested at 150 °C [302 °F]

where n and A are constants.

Thompson and Odegard (1973) also calculated the activation energy, Q, for a given uniaxial stress, σ, using the following empirical equation

$$Q = -R\left[\frac{\Delta \ln \dot{\varepsilon}(\varepsilon)}{\Delta\left(1/T\right)}\right] \tag{2-8}$$

where R is the universal gas constant; σ is the constant uniaxial normalized stress with respect to the corresponding yield stress at a given temperature, T; and $\dot{\varepsilon}$ is the strain rate at a selected strain level, ε, at which the activation energy, Q, is calculated.

In this investigation, Titanium Grade 7 specimens were creep tested at various temperatures—25, 50, 100, and 150 °C [77, 122, 212, and 302 °F], at 85 percent of the respective yield stress at these temperatures. The creep curves for these tests are shown in Figure 2-10.

The unexpected result from this investigation is that the creep strain does not necessarily increase as temperature increases, as is expected for metallic materials. While the creep strain does increase with temperature for the earliest stages of creep (up to about five hours), the

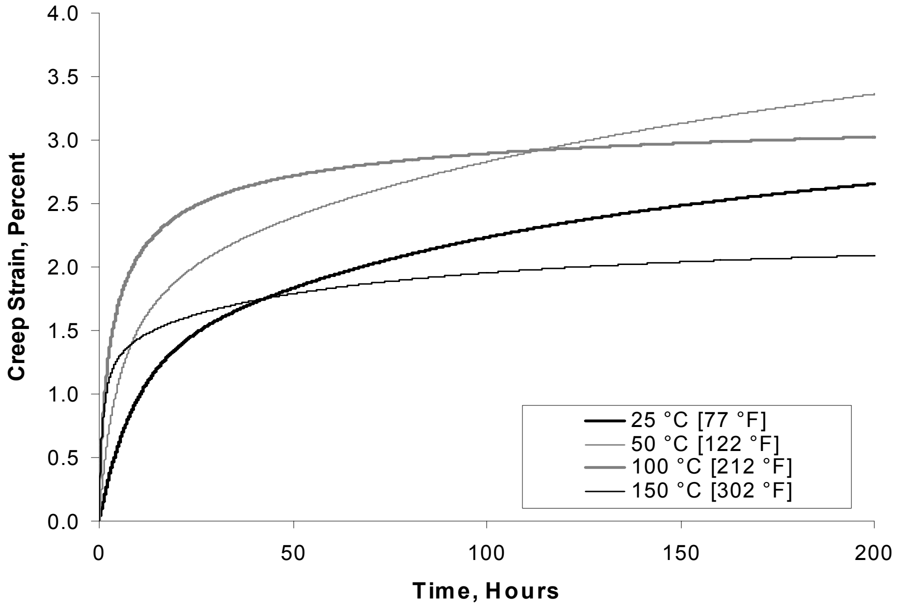


Figure 2-10. Creep Curves for Titanium Grade 7 Tested at 25, 50, 100, and 150 °C [77, 122, 212, and 302 °F] at 85 Percent Yield Stress

creep strain rate begins to decrease rapidly at temperatures above 50 °C [122 °F] at the later stages of creep. This suggests that a creep deformation mechanism may not be thermally activated, because if the mechanism is thermally activated, creep strain rate will always increase with temperature.

To help identify the active creep deformation mechanism, the activation energy for creep was measured at various strain levels. This process involves measuring the instantaneous creep strain rate, $\dot{\varepsilon}$, at various strain levels for each temperature. When the natural logarithm of instantaneous creep strain rate is plotted as a function of the reciprocal of temperature, a line is obtained with a slope proportional to the activation energy (by a factor of $-R$) for creep at a given strain level.

Consider first the earliest stages of creep where the creep strain rate does increase with temperature. At three different strain levels (0.1 percent, 0.2 percent, and 0.4 percent), the instantaneous creep strain rate was determined at the various temperatures. Figure 2-11 shows the plot of the natural logarithm of the instantaneous creep strain rate versus reciprocal temperature for these strain levels. First note that the instantaneous creep strain rate for the specimens tested at 100 and 150 °C [212 and 302 °F] are nearly the same. However, the strain is increasing so rapidly at this point that it is difficult to get a precise measurement of the instantaneous creep strain rate at these strain levels {for instance, at 150 °C [302 °F], the strain is already at about 0.16 percent after the first measurement and about 0.28 percent after the second measurement}. Due to this uncertainty, a best-fit line to determine the activation energy at these strain levels was drawn only from the data taken at 25, 50, and 100 °C [77, 122, and 212 °F]. The empirical equation for the respective best-fit lines is shown in Figure 2-11. To

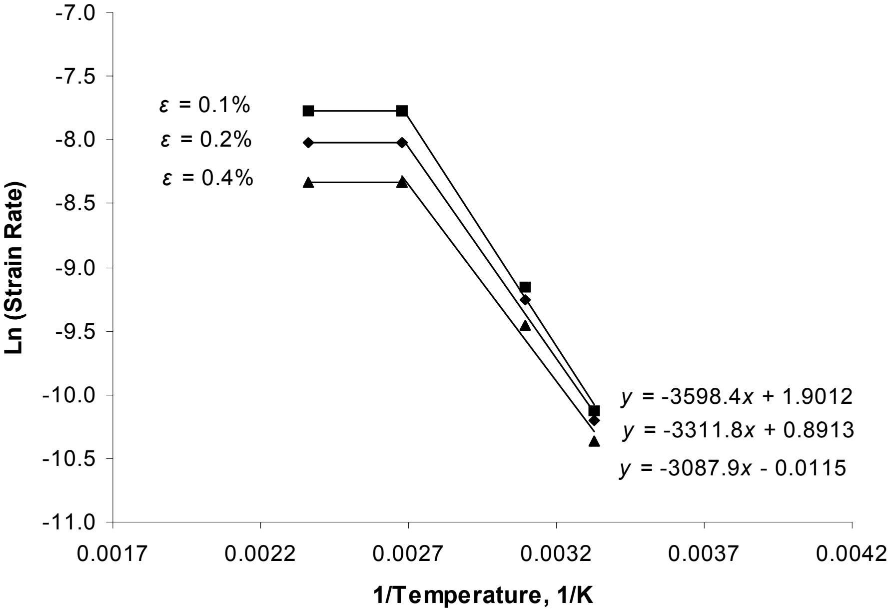


Figure 2-11. Natural Logarithm of the Instantaneous Creep Strain Rate Versus Reciprocal Temperature for Grade 7 Titanium at Strain Levels of 0.1, 0.2, and 0.4 Percent

determine the activation energy for creep at these strain levels, the slope of the best fit line is multiplied by $-R$ {-8.31 J × K^{-1} × mol^{-1} [-1.99 cal × K^{-1} × mol^{-1}]}. These values are shown in Table 2-2. The measured activation energy of about 30 kJ/mol [7.17 kcal/mol] is consistent with previously measured values for the activation energy for creep of α-titanium when slip is the predominant creep deformation mechanism (Tung and Sommer, 1970; Zeyfang, et al., 1971). Slip is a thermally activated process (slip is easier as temperature increases), which explains the increase in creep strain rate as temperature increases during the earliest stages of creep.

The fact that the creep strain rate of the specimens tested at the elevated temperature begins to decrease compared to those of the specimens tested at the lowest temperatures suggests that after the earliest stages of creep, another athermal (not thermally activated) creep deformation mechanism is operative. One such deformation mechanism is twinning, which is a common deformation mechanism in titanium alloys. The critical stress for twinning shows little temperature dependence, and twinning becomes more common as temperature decreases (Song and Gray, 1995a; Meyers, et al., 2001). This is because at high temperatures, the critical stress for slip is less than the critical stress for twinning. Thus, slip is the predominant deformation mechanism. As temperature decreases, the critical stress for slip increases while the critical stress for twinning remains nearly constant. When the critical stress for slip becomes higher than the critical stress for twinning, twinning will become the predominant deformation mechanism. Twinning activity at lower temperatures could explain the continued creep at these temperatures, whereas the higher temperature specimens cannot creep by twinning.

Table 2-2. Activation Energies for Creep of Titanium Grade 7 at 85 Percent Yield Stress at Various Strain Levels

Strain Level, Percent	Activation Energy kJ/mol [kcal/mol]
0.1	29.9 [7.14]
0.2	27.5 [6.57]
0.4	25.7 [6.14]
1.0	29.1 [6.96]
1.5	35.2 [8.41]
2.0	30.0 [7.17]
2.5	30.0 [7.17]

While twinning initiation may be an athermal process, twins must grow for creep strain to proceed, and twin growth is a thermally activated process. It has been shown that twin growth is rate-limited by the diffusion of oxygen due to the nonconservation of octahedral interstitial lattice where oxygen atoms can reside (Oberson and Ankem, 2005). Thus, for the temperature range where twinning is an active deformation mechanism, the twin growth rate will increase with increasing temperature, and the activation energy for creep should correspond to the activation energy for the diffusion of oxygen in titanium. Therefore, the method of Thompson and Odegard (1973) can be used to calculate the activation energy for creep at higher strain levels only at 25 and 50 °C [77 and 122 °F]. This means that the plot of the natural logarithm of instantaneous creep strain rate versus reciprocal temperature will only have two points from which to draw a best-fit line. Having more data points from which to derive this line (if tests had been performed at other temperatures where twinning was an active deformation mechanism) would provide a more reliable measurement of the activation energy.

For the specimens tested at 25 and 50 °C [77 and 122 °F], the instantaneous creep strain rate was measured at the stress levels of 1.0, 1.5, 2.0, and 2.5 percent, and the natural logarithm of the instantaneous strain rate was plotted versus reciprocal temperature, as shown in Figure 2-12. The slope of the best-fit line connecting the two data points was multiplied by $-R$ to calculate the activation energy for creep at the various strain levels. These values are shown in Table 2-2. The value of the activation energy for creep measured at the higher strain level is slightly above that measured for low strain, as seen in the plot in Figure 2-13, suggesting that there could be a change in the predominant deformation mechanism from solely slip at low strain to slip and twinning at higher strain. The activation energy for the diffusion of oxygen in α-titanium is in the range of 65 to 200 kJ/mol [15.5 to 47.8 kcal/mol] (Liu and Welsch, 1988), which is somewhat above the measured activation energy for creep in this investigation. That is because at the strain levels from 1 to 2.5 percent slip and twinning are simultaneous deformation mechanisms for Titanium Grade 7 and the overall activation energy for creep will be closer to that of slip than twinning. If creep strain was to proceed further, twinning would become predominant compared to slip and the activation energy for creep would increase to higher values approaching those of the activation energy for the diffusion of oxygen in titanium.

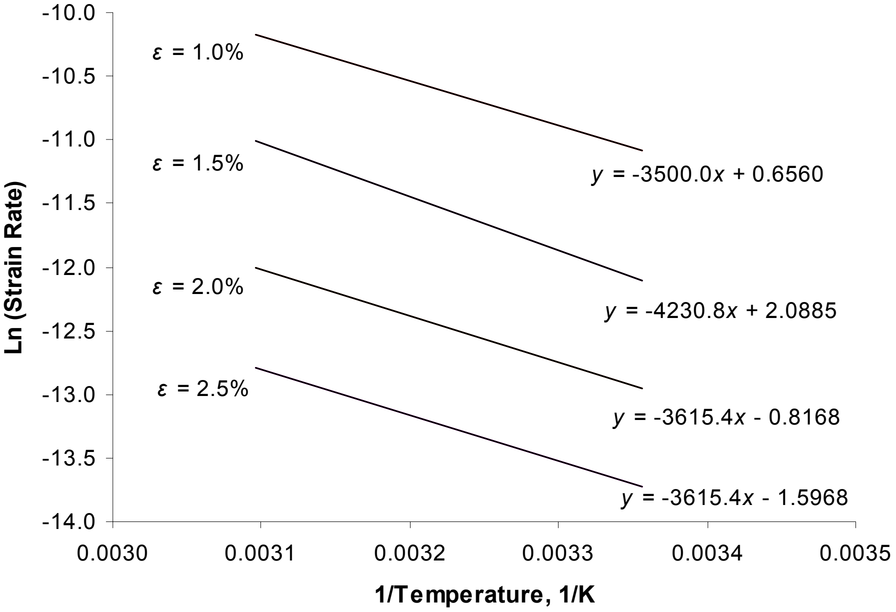


Figure 2-12. Natural Logarithm of the Instantaneous Creep Strain Rate Versus Reciprocal Temperature for Grade 7 Titanium at Strain Levels of 1.0, 1.5, 2.0, and 2.5 Percent

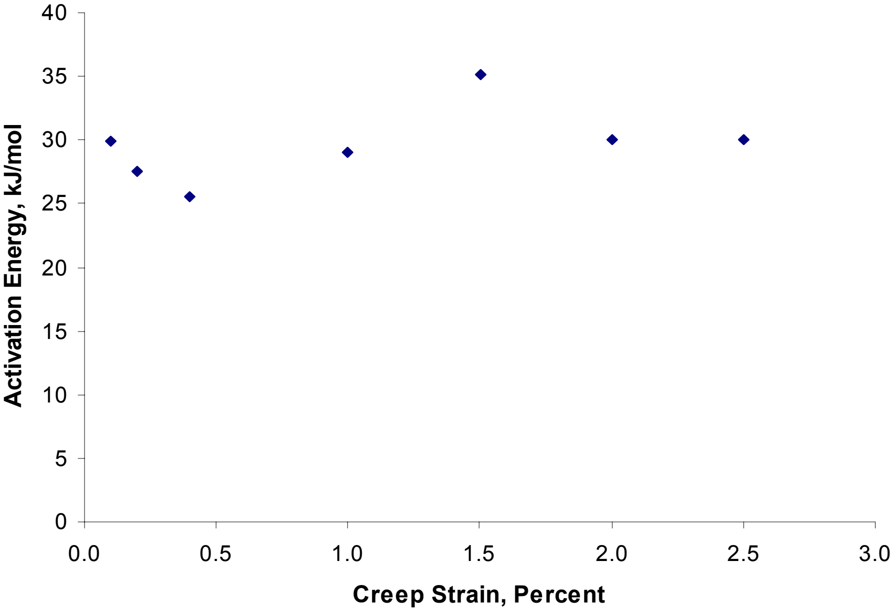


Figure 2-13. Activation Energy for Creep of Grade 7 Titanium Versus Creep Strain Level

The other factor that must be considered for the decrease in creep strain with increasing temperature is the way the applied stress level was determined. It is conventional to perform the creep test at a known fraction of the 0.2 percent yield stress. Due to the lack of a clear transition from linear elastic to plastic strain, the yield stress can be defined as the stress at some arbitrary plastic strain (typically 0.2 percent). In most cases, the stress flattens off after yielding or increases due to strain hardening. Thus, the 0.2 percent yield stress will usually be the same as or slightly higher than the actual stress at which instantaneous plastic deformation begins. This is the case for Titanium Grade 7 tested at 25 and 50 °C [77 and 122 °F]. However, for Titanium Grade 7 tested at 100 °C [212 °F] and 150 °C [302 °F], there is a significant decrease in stress after a peak stress is reached at the end of the linear elastic portion of the stress-strain curve. When the 0.2 percent yield stress is determined at 100 °C and 150 °C [212 and 302 °F], it is somewhat smaller than the peak stress at which instantaneous plastic deformation will begin (see Appendix A). Thus, when the applied stress level is 85 percent of the 0.2 percent yield stress at 100 and 150 °C [212 and 302 °F], instantaneous plastic deformation begins at a smaller percentage of the actual stress level. This could contribute to decreased creep strain at the elevated temperatures compared to the lower temperatures.

2.2 Microscopy—Undeformed Titanium Grade 7

An undeformed, as-received plate of Titanium Grade 7 was examined by optical microscopy to characterize the microstructure testing in terms of grain size and rolling texture.

2.2.1 Procedures

2.2.1.1 Hand Polishing

The goal of polishing is to obtain a smooth and scratch-free surface with no deformation products such as twins. The first step is to hand polish the surfaces of the specimen with silicon carbide grit paper. Relatively coarse 600-grit paper is used first, followed by 800- and then 1,200-grit paper. With each successively finer grit, polishing is done perpendicularly to that of the previous grit polishing until no scratches are visible to the naked eye. The specimen is rinsed with water and dried with compressed air every few minutes to reduce abrasion by residual particles. The 600-grit paper was used for approximately 10 minutes per side followed by 30 minutes per side for the 800 and 1,200 grit (time that is necessary to remove scratches when polishing at a moderate speed). Colloidal silica polishing suspension on a soft pad was used for the final polishing of the creep specimens. The specimen was polished for approximately 20 minutes on each side to achieve a mirrorlike finish and until no scratches or surface features were visible under an optical microscope.

2.2.1.2 Etching

Chemical etching is done to reveal the grain boundaries. Two different etchants are used: Kroll’s reagent and R-etch (the composition of these etchants is shown in Tables 2-3 and 2-4, respectively). The specimen surface is etched very lightly with Kroll’s reagent for approximately 20 seconds per side with a cotton swab. Kroll’s reagent is a low-contrast etchant. Then the specimen is rinsed with water and etched with R-etch for approximately 10 seconds to stain the surface of the specimen.

Table 2-3. Chemical Composition of Kroll's Reagent	
Chemical	**Amount, mL [fl. oz.]**
Hydrofluoric Acid (HF 50%)	2 [0.068]
Nitric Acid (HNO_3)	10 [0.34]
Distilled Water (H_2O)	88 [2.98]

Table 2-4. Chemical Composition of R-Etchant	
Chemical	**Amount**
Hydrofluoric Acid (HF 50%)	25 mL [0.85 fl. oz.]
Benzalkonium Chloride	18.5 g [0.65 oz.]
Glycerine	40 mL [1.35 fl. oz.]

2.2.1.3 Microscopy

Optical micrographs of the as-received specimen in the Longitudinal-Transverse (L-T), Short-Transverse (S-T), and Short-Longitudinal (S-L) orientations, as shown in Figure 2-14. An inverted metallographic light microscope was used for optical microscopy.

2.2.1.4 Average Grain Size Calculations

The average grain size for the as-received Titanium Grade 7 specimen was determined using ASTM Standard E112-96, “Standard Test Methods for Determining Average Grain Size” (ASTM International, 2007a). According to the lineal intercept procedure, the average grain size is estimated by counting the number of grains intercepted by one or more straight lines. Counts are made on several widely separated and random fields to obtain an average value for the specimen. Where the grain shape is altered by the process such that the grains are no longer equiaxed, as in this case, measurements are made on the L-T, S-L, and S-T planes.

2.2.2 Results

2.2.2.1 Optical Micrographs

The optical micrographs of the various orientations for the as-received Titanium Grade 7 specimen are shown on Figure 2-15. The grains appear to be equiaxed, showing little evidence of elongation in the rolling direction.

2.2.2.2 Average Grain Size Calculations

The average grain sizes for the respective orientations were calculated as described in Section 2.2.1.4, and are given in Table 2-5.

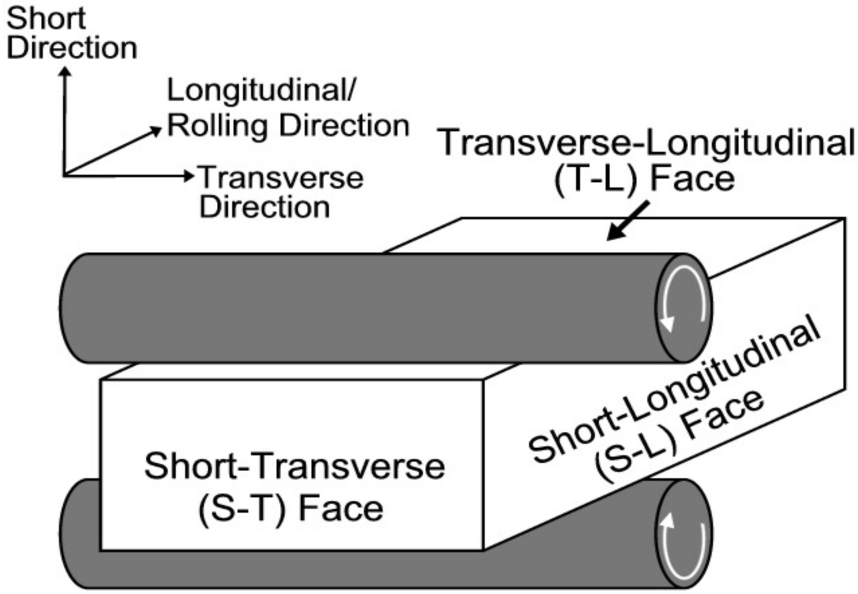


Figure 2-14. Schematic Illustration of As-Received Titanium Grade 7 Plate

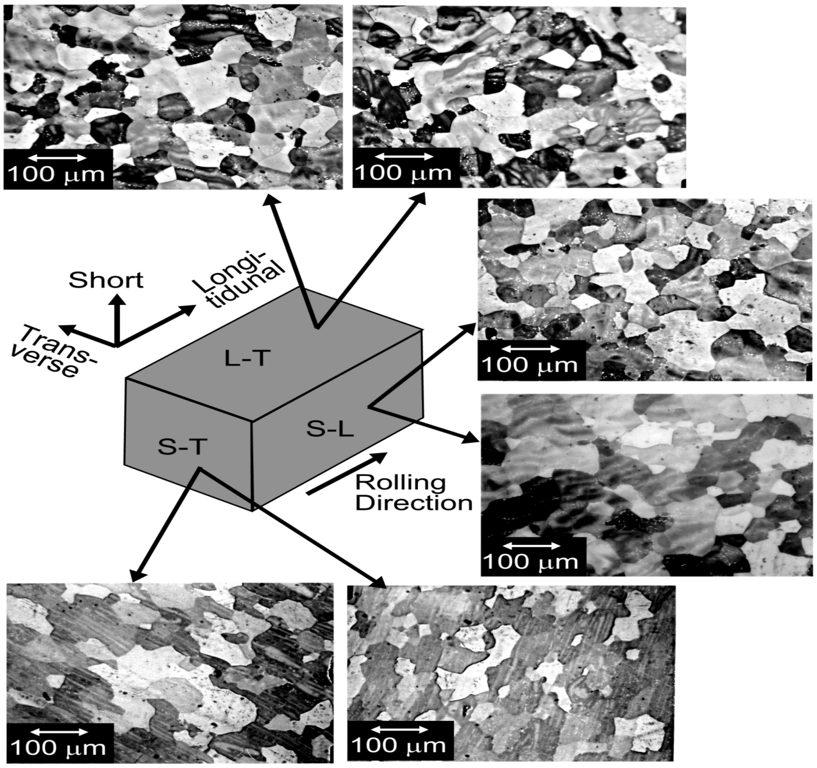


Figure 2-15. Optical Micrographs of As-Received Titanium Grade 7 With a Horizontal Rolling Direction in All Micrographs

Table 2-5. Average Grain Size for As-Received Titanium Grade 7

Orientation	Average Grain Size, μm [mil]
Longitudinal-Transverse (L-T)	55 [2.17]
Short-Transverse (S-T)	52 [2.05]
Short-Longitudinal (S-L)	59 [2.32]

2.3 Transmission Electron Microscopy

Transmission electron microscopy is a high-magnification imaging technique whereby an electron beam is passed through a material. Transmission electron microscopy can be used to characterize the crystallographic structure of a material and identify deformation products in the creep-tested material (Williams and Carter, 1996).

2.3.1 Procedures

2.3.1.1 Transmission Electron Microscopy Sample Preparation

The success of transmission electron microscopy depends on the specimens that must remain unaltered and be sufficiently thin for electron transparency. The first step is a removal of a specimen from the bulk material. A low-speed saw with a diamond wafering blade is used to slice sections of material from the creep specimens. One slice is made initially to reveal the interior of the specimen, then subsequent slices are made by moving the sample holder a set distance in relation to the blade. This is accomplished by dialing in the attached micrometer to the desired displacement. Eighteen divisions of the micrometer yield approximately 120-mm [0.045-in]-thick specimens. A disk punch is then used to produce 3-mm [0.1181-in]-diameter disks from the cut slices. The punch minimizes additional deformation into the specimen.

The next step involves prethinning the transmission electron microscopy disks by dimpling, which mechanically polishes a divot or dimple into the center of the disk. A specimen stage is heated on a hot plate, and a small amount of adhesive is melted onto the top of the stage to adhere the disk to the stage. The disk is placed as close as possible to the center of the stage inside the centering ring, and the stage is removed from the hot plate and allowed to cool for about 10 minutes. Once cooled, the stage is placed onto the dimpling apparatus underneath the polishing wheel. A small amount of 6-μm [0.24-mil] diamond paste is placed on top of the specimen and on the polishing wheel. The specimen is then dimpled until the diameter of the dimple is approximately 2 mm [0.0787 in]. This corresponds to a dimple depth of approximately 40 μm [0.0016 in]. The stage is then removed from the instrument and heated on the hot plate to release the specimen. The specimen is flipped over and the process is repeated on the other side, leaving the thickness at the center of the specimen about 40 μm [0.0016 in].

Following dimpling, the final thinning was performed by jet polishing. Jet polishing is a chemical process that uses a combination of an acid containing electrolyte and electric current to remove material from the sample. Specimens were prepared in a twin-jet polishing unit. The polishing solution used for jet polishing consists of 3 percent hydrochloric acid, 3 percent sulfuric acid, and 94 percent methanol. The dish containing the polishing solution is placed in a methanol bath and cooled with dry ice so that the samples are polished in the range of −60 to −70 °C [−140 to −158 °F] at 20 V.

2.3.1.2 Transmission Electron Microscopy Operations

Two different transmission electron microscopes have been used in this investigation: a JEOL 2100F Field Emission Transmission Electron Microscope and a JEOL 2100 LaB_6, both operating at 200 keV. A combination of techniques is needed to understand the deformation mechanisms, including selected area diffraction, bright field imaging, and dark field imaging.

The various deformation mechanisms were identified in several ways. Slip was identified by standard techniques (Williams and Carter 1996): dark field imaging using three different g (reciprocal lattice) vectors and $g \cdot b$ (the dot product of $g \cdot$ and b) analysis where b is the Burgers vector of the dislocations. To determine whether slip involving screw dislocations with a Burgers vector, $\vec{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$, or *a*-type dislocations, was gliding on basal or prism planes, bright and dark field images were taken along $\langle 11\bar{2}0 \rangle$ and $[0001]$ zone axes and tilted slightly to determine whether the visible dislocation lines were single dislocations or stacks of dislocations on single planes in projection. This was necessary because dislocations in hexagonal crystals can glide on basal or prism slip planes.

To identify twins, selected area diffraction patterns were taken from the matrix and across the twin matrix interface. To identify a twin, the specimen must be tilted along a zone axis that contains the g vector for the twin plane common to both the twin and the matrix.

2.3.2 Undeformed Titanium Grade 7

Undeformed Titanium Grade 7 transmission electron microscopy specimens were sectioned from the threaded ends of a creep-tested specimen and prepared as described in Section 2.3.1.1. The creep deformation is localized in the narrow gage length. Undeformed specimens were examined to confirm the crystal structure of the material and to look for evidence of any deformation products, such as dislocations, which may be present prior to creep testing.

The transmission electron microscopy of undeformed specimens only shows grain boundaries and occasional groups of a few dislocations, as seen on Figure 2-16. These are expected as a low density of dislocations to be present in any metal or alloy. The undeformed specimens, however, serve as a baseline to which the creep deformed specimens can be compared. Figure 2-17 is an example of a selected area diffraction pattern taken from the undeformed specimen. The image is taken along the $\langle 1\bar{2}13 \rangle$ zone axis, confirming that the material has the hexagonally close-packed crystal structure.

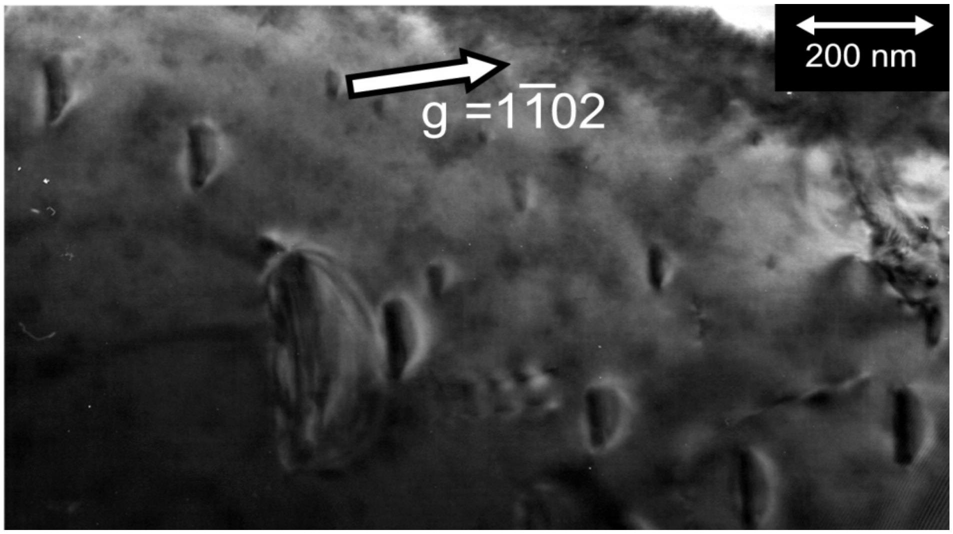


Figure 2-16. Bright-Field Transmission Electron Micrograph of a Group of Dislocations in the Undeformed Titanium Grade 7 Specimen. Note: In This and the Following Transmission Electron Micrographs, the Arrow Refers to the $\vec{g}$ Vector, the Indices of Which Are Given.

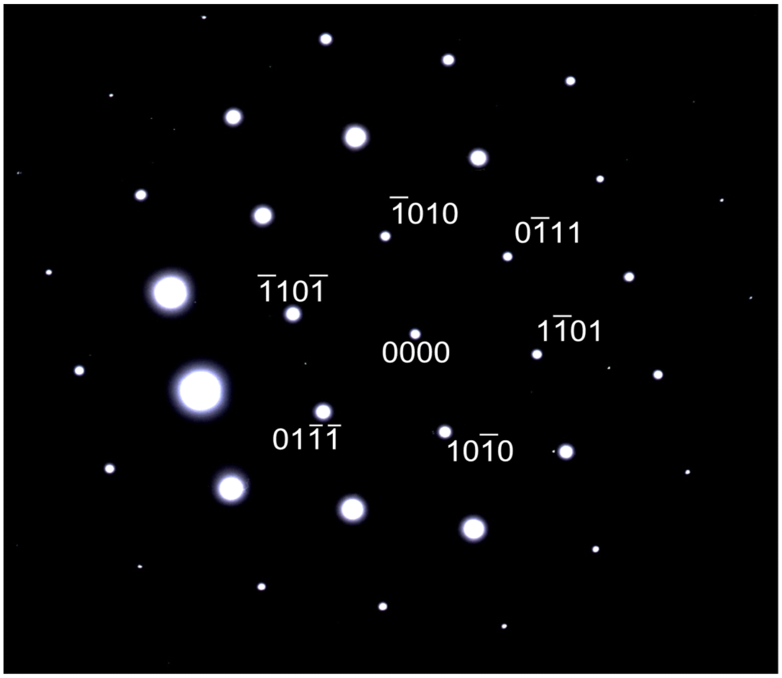


Figure 2-17. Selected Area Diffraction Pattern of Undeformed Titanium Grade 7 Taken Along the $\langle 1\bar{2}13 \rangle$ Zone Axis

2.3.3 Creep Deformed Titanium Grade 7

The creep deformed Titanium Grade 7 specimens tested at various stress levels at a constant temperature of 150 °C [302 °F] and at different temperatures at a constant stress level of 85 percent yield stress were examined by transmission electron microscopy to identify the deformation mechanisms (i.e., slip and twinning). Specimens were sectioned from the deformed region of the creep-tested gage length.

2.3.3.1 Transmission Electron Microscopy of Specimens Tested at 150 °C [302 °F]

2.3.3.1.1 Specimen Tested at 100 Percent Yield Stress

The specimen tested at 100 percent yield stress deformed to a creep strain of approximately 13.7 percent after 200 hours. In comparison to the undeformed specimen (Figure 2-16), there are a significant number of deformation products in the creep-deformed specimen. The transmission electron microscopy investigation shows that there are two predominant deformation mechanisms in this specimen: slip and deformation twinning. Figure 2-18(a) and (b) show dislocation arrays of *a*-type screw dislocations, which have Burgers vectors of the type $\vec{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$ and glide on prism $\{1\bar{1}00\}$ planes. This is the most common type of dislocation that is active during creep deformation of hexagonally close-packed materials, particularly given the observed crystallographic texture and loading orientation (Zeyfang, et al., 1971; Conrad, et al., 1973; Paton, et al., 1973; Neeraj, et al., 2000).

The second predominant deformation product observed in the transmission electron microscopy examination is deformation twins. Twinning is a deformation mechanism in which the atoms in the twinned part of the material are sheared to positions that form a mirror with that of the untwinned part of the materials (Christian and Mahajan, 1995). Twins have long been associated with tensile deformation in α-titanium (Partridge, 1968; Conrad, et al., 1973; Akhtar, 1975; Yoo, 1981; Christian and Mahajan, 1995; Song and Gray, 1995a,b; Meyers, et al., 2001), although some studies have identified deformation twinning during low temperature creep of α-titanium alloys (Ankem, et al., 1994; Hultgren, et al., 1999; Aiyangar, et al., 2005).

According to the classical theory of deformation twinning (Christian and Mahajan, 1995), the original untwinned (matrix) lattice is transformed by displacements that are equivalent to or an integral fraction of a simple shear of the lattice points. There are two planes which are not distorted by the shear of the lattice. One of these, denoted K_1, is referred to as the twinning plane, and the shear direction is given by η_1. The second undistorted, or conjugate plane is denoted K_2, and the plane of shear, P, contains η_1 and the normals to K_1 and K_2. The intersection of K_2 and P gives the conjugate or reciprocal shear direction, η_2. When describing a twin, it is typical to identify the twin by the shear plane K_1 and the shear direction η_1 (i.e., $\{\mathrm{hkil}\}\langle \mathrm{uvtw} \rangle$) or more commonly by only the shear plane.

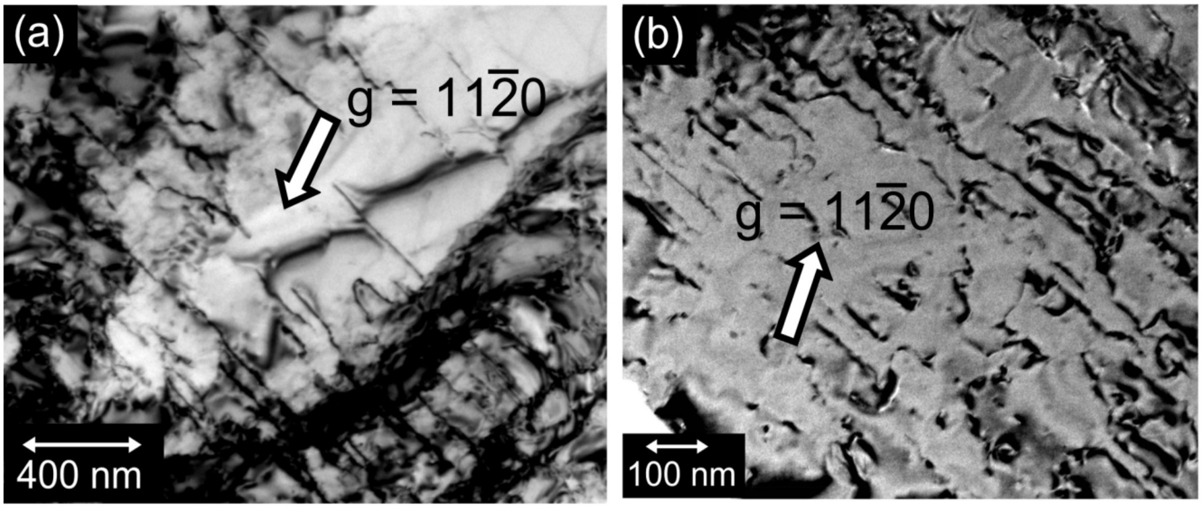


Figure 2-18. Bright-Field Transmission Electron Micrographs of Arrays of *a*-Type Prism Dislocations in Titanium Grade 7 Creep Deformed at 100 Percent Yield Stress at 150 °C [302 °F]

Alternatively, the twinned lattice can be related to the untwinned lattice by a rotation around a pole perpendicular to both the twin plane normal and the twinning direction. As such, when using transmission electron microscopy, twins are identified by obtaining selected area diffraction patterns of the matrix and the twin where the zone axis is the direction of the pole of rotation. The matrix and twin will have the same selected area diffraction pattern, but they will be rotated with respect to one another at an angle that is characteristic of the twinning mode. There are four main twinning modes which have been identified in hexagonal close-packed materials (Partridge, 1968). The modes are characterized by a particular twinning plane (K_1) and direction (η_1).

In the case of creep-deformed Titanium Grade 7 in this investigation, the twinning mode was identified as $\{1\bar{1}02\}\langle 10\bar{1}\bar{1}\rangle$, hereafter to be referred to as the $\{1\bar{1}02\}$ type twin. This is the most common twinning mode in α-titanium alloys (Song and Gray, 1995b), given the observed crystallographic texture and loading orientation (see Appendix B). In this case, the twin and the matrix are related by a rotation of approximately 85° along the shear plane normal. Figure 2-19, shows the untwinned matrix, a $\{1\bar{1}02\}$ twin, and across the twin–matrix interface. The selected area diffraction patterns of the $\{\bar{1}\bar{1}20\}$ foil plane taken from the area are presented in Figure 2-20. Note that the diffraction pattern of the twin–matrix interface is symmetric with respect to the line that passes through the shared $\{1\bar{1}02\}$ spots, which is indicated by the dashed line. Similar $\{1\bar{1}02\}$ twins were found throughout the specimen.

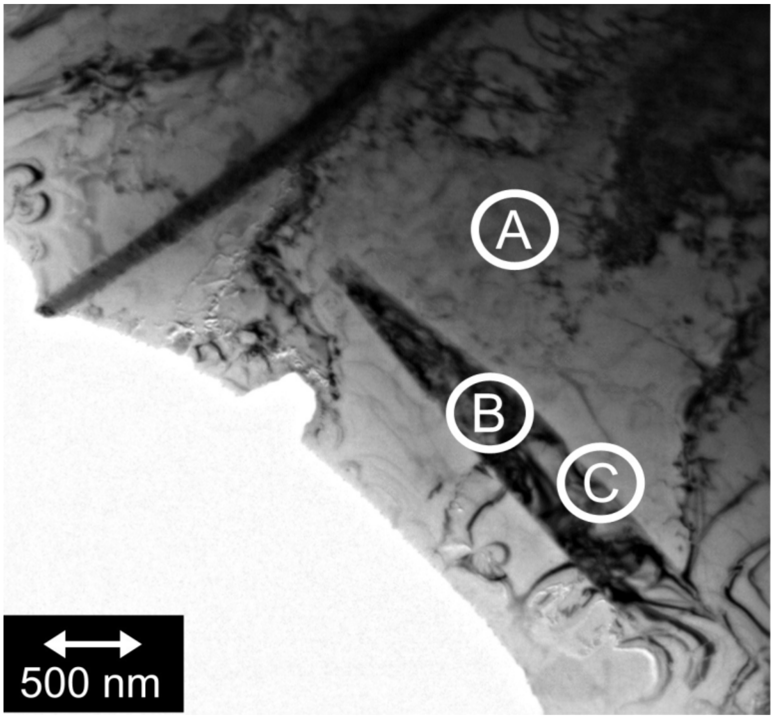


Figure 2-19. Bright-Field Transmission Electron Micrograph of $\{1\bar{1}02\}$ Twin Showing the Areas From Which the Diffraction Patterns in Figure 2-20 Were Taken. "A" Is the Untwinned Matrix, "B" is Inside the Twin, and "C" is Across the Twin–Matrix Interface.

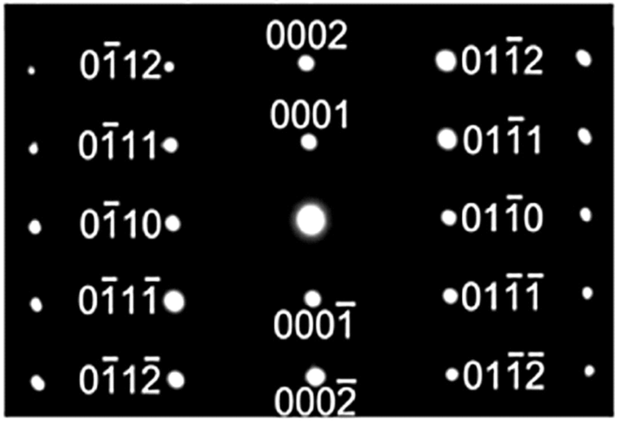
2.3.3.1.2 Specimen Tested at 85 Percent Yield Stress

For the Titanium Grade 7 specimen creep tested at 150 °C [302 °F] at 85 percent yield stress, the creep strain after 200 hours was approximately 2.1 percent. For this specimen, the only observed deformation mechanism is *a*-type prism slip, $\vec{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$, gliding on prism $\{1\bar{1}00\}$ planes. The dislocations are frequently observed in the aligned arrays as seen in Figure 2-21. There are no deformation twins observed in this specimen as there were in the specimen tested at 100 percent yield stress. It is likely that given the limited strain of the 85 percent yield stress, there is insufficient stress concentration for twin nucleation, particularly if twins are nucleated by dislocation pileups (Song and Gray, 1995a).

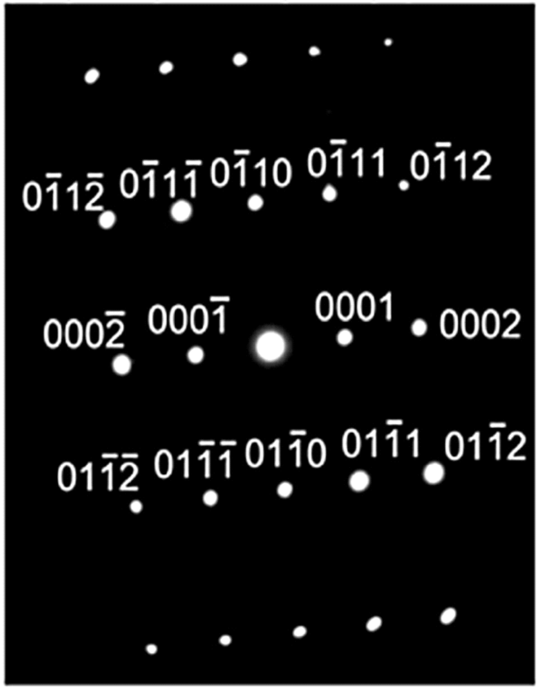
2.3.3.1.3 Specimen Tested at 70 Percent Yield Stress

For the Titanium Grade 7 specimen creep tested at 150 °C [302 °F] at 70 percent yield stress, the creep strain after 200 hours is approximately 0.39 percent. As for the specimen tested at 85 percent yield stress, the only observed deformation mechanism is *a*-type prism slip, $\vec{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$, gliding on prism $\{1\bar{1}00\}$ planes. There is no twinning observed in this specimen,

(a) Matrix: $\{\bar{1}\bar{1}20\}$ plane



(b) $\{1\bar{1}02\}$ Twin: $\{\bar{1}\bar{1}20\}$ plane



(c) $\{1\bar{1}02\}$ Twin: Twin-Matrix interface

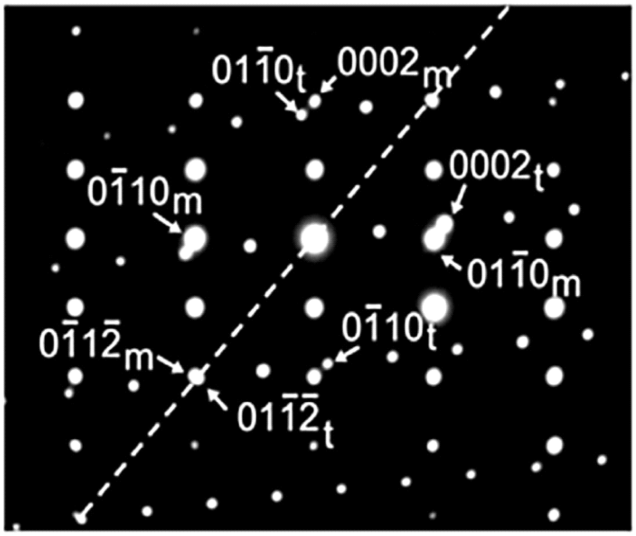


Figure 2-20. Selected Area Diffraction Patterns Along the $\langle\bar{1}\,\bar{1}20\rangle$ Zone Axis Taken From Creep Deformed Titanium Grade 7, Shown in Figure 2-19. (a) Untwinned Matrix; (b) $\{1\bar{1}02\}$ Twin. Diffraction Pattern Is Rotated Approximately 85° Clockwise With Respect to the Untwinned Matrix. (c) Across Twin–Matrix Interface. Spots With the Subscript "m" refer to Matrix Spots, and Spots With the Subscript "t" Refer to Twins Spots.

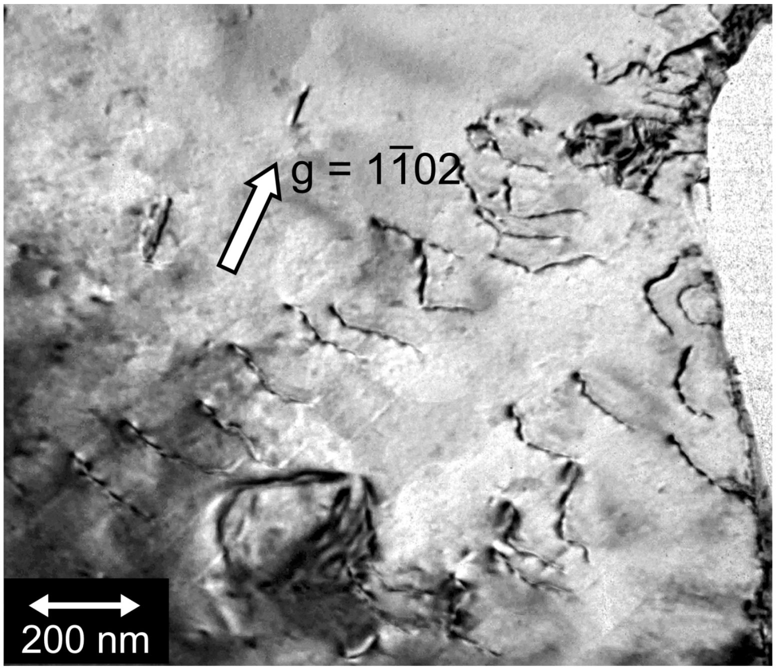


Figure 2-21. Bright-Field Transmission Electron Micrograph of *a*-Type Prism Dislocation Array in Titanium Grade 7 Creep Deformed at 85 Percent Yield Stress at 150 °C [302 °F]

which is expected, given the limited strain for this testing condition. At the low strain level, dislocation arrays are difficult to find, but examples are shown in Figure 2-22.

2.3.3.1.4 Specimens Tested at 55 Percent Yield Stress and 40 Percent Yield Stress

Given the extremely limited strain of the Titanium Grade 7 specimens creep tested at 55 and 40 percent yield stress (approximately 0.17 percent and 0.05 percent, respectively), these specimens were not examined with transmission electron microscopy. It would be very difficult to identify and characterize the deformation mechanisms. It is likely, however, that as is the case for the specimens tested at 85 and 70 percent yield stress, *a*-type prism slip is the predominant deformation mechanism.

2.3.3.2 Transmission Electron Microscopy of Specimens Tested at 85 Percent Yield Stress

2.3.3.2.1 Specimen Tested at 150 °C [302 °F]

Results for the specimen tested at 85 percent yield stress at 150 °C [302 °F] are presented in Section 2.3.1.1.2.

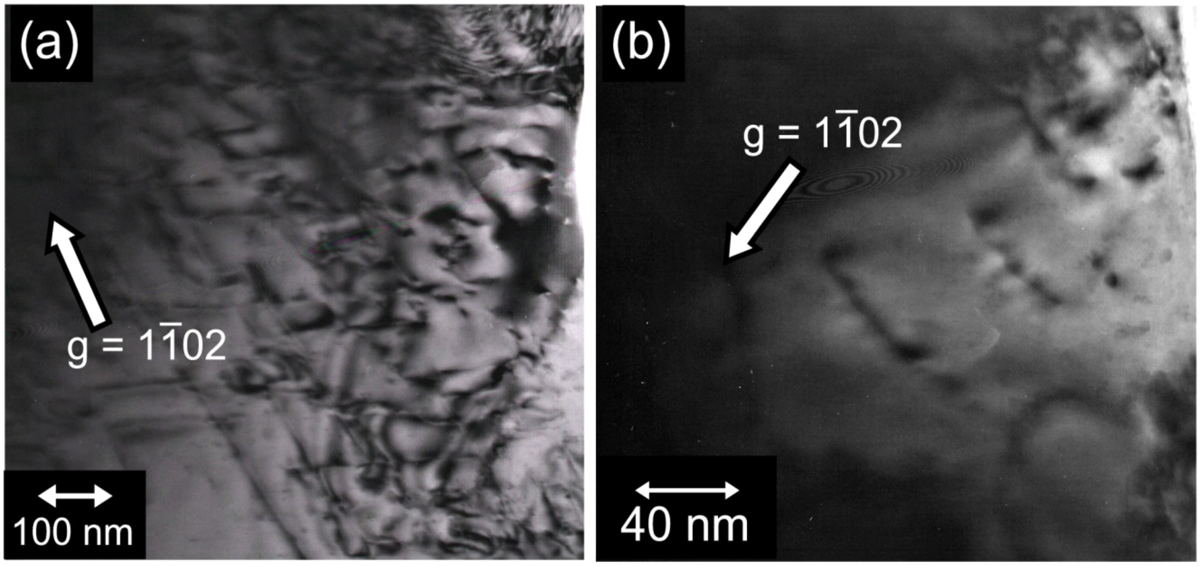


Figure 2-22. Bright-Field Transmission Electron Micrograph of *a*-Type Prism Dislocations in Titanium Grade 7 Creep Deformed at 70 Percent Yield Stress at 150 °C [302 °F]

2.3.3.2.2 Specimen Tested at 100 °C [212 °F]

The Titanium Grade 7 specimen creep tested at 100 °C [212 °F] at 85 percent yield stress had a creep strain of approximately 3.02 percent after 200 hours. For this specimen, the only observed deformation mechanism was *a*-type prism slip, $\vec{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$, gliding on prism $\{1\bar{1}00\}$ planes, as seen in Figure 2-23. No deformation twinning was seen in this specimen. This behavior is similar to that observed at 150 °C [302 °F].

2.3.3.2.3 Specimen Tested at 50 °C [122 °F]

The Titanium Grade 7 specimen creep tested at 50 °C [122 °F] at 85 percent yield stress had a creep strain of approximately 3.36 percent after 200 hours. For this specimen, slip and occasional twinning were observed to be creep deformation mechanisms, as seen in Figures 2-24 (a) and (b), respectively. The slip was identified as *a*-type prism slip, $\vec{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$, gliding on prism $\{1\bar{1}00\}$ planes, and the twins were identified as $\{1\bar{1}02\}$ type. It is not unexpected that twins were found in this specimen and not in those tested at 85 percent yield stress at higher temperatures (Song and Gray, 1995b). This is because twinning is generally favored at lower temperatures as a result of the critical stress for dislocation glide increasing while temperature decreases and the critical stress for twinning remains constant and independent of temperature. As such, at lower temperatures, it is more likely that the critical stress for twinning will be reached before that of slip.

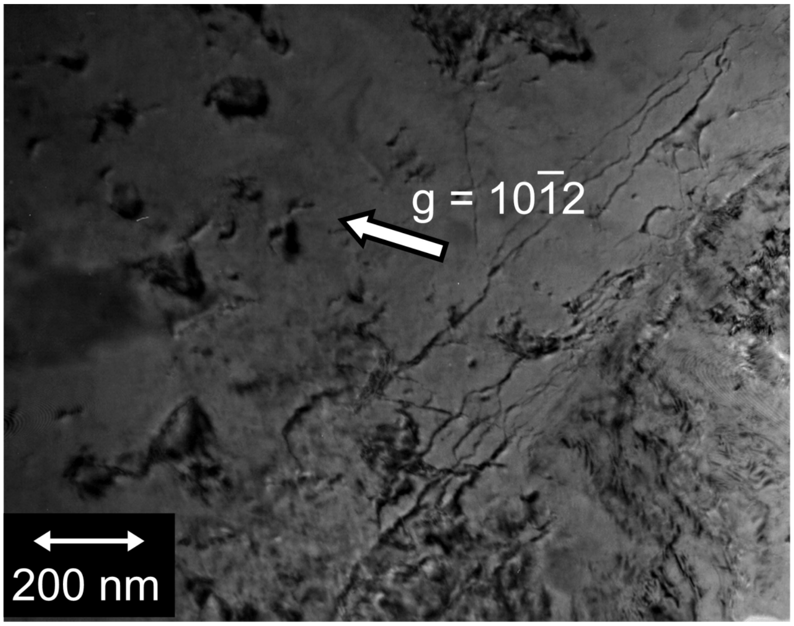


Figure 2-23. Bright-Field Transmission Electron Micrograph of *a*-Type Prism Dislocations in Titanium Grade 7 Creep Tested at 85 Percent Yield Stress at 100 °C [212 °F]

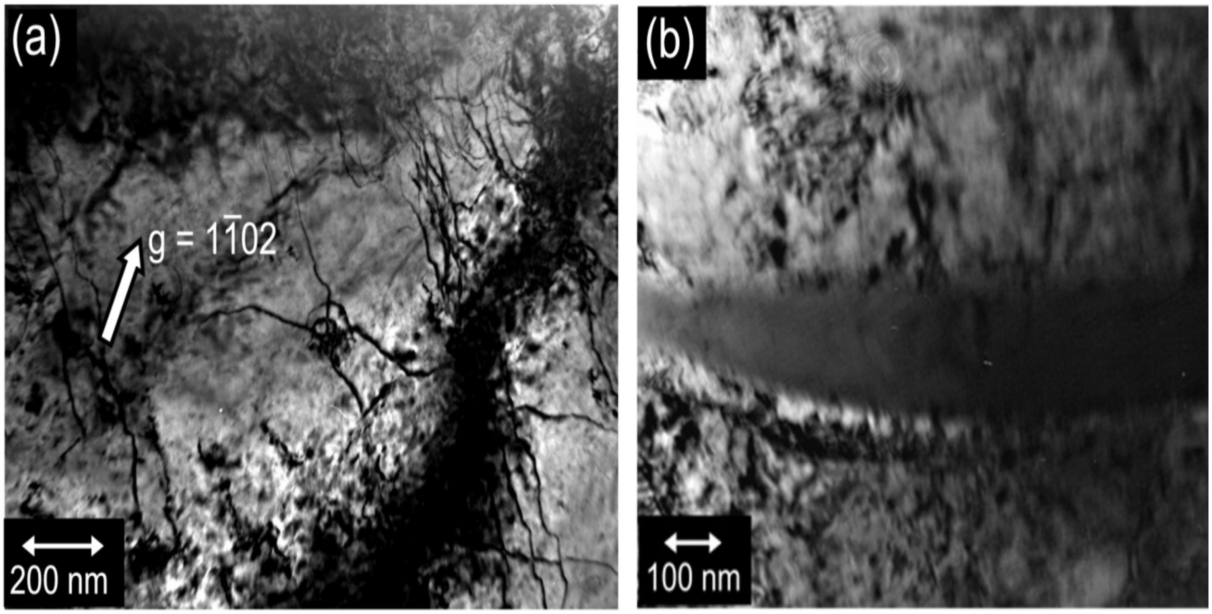


Figure 2-24. Bright-Field Transmission Electron Micrographs of Titanium Grade 7 Creep Tested at 50 °C [122 °F] at 85 Percent Yield Stress: (a) *a*-Type Prism Dislocations and (b) $\left\{1\bar{1}02\right\}$ **Type Twin**

2.3.3.2.4 Specimen Tested at 25 °C [77 °F]

The Titanium Grade 7 specimen creep tested at 25 °C [77 °F] at 85 percent yield stress had a creep strain of approximately 2.65 percent after 200 hours. For this specimen, as for the one tested at 50 °C [122 °F], slip and twinning were identified as active creep deformation mechanisms, as seen in Figure 2-25. Twinning was more common at 25 °C [77 °F] than at 50 °C [122 °F], which was expected. The slip was identified as *a*-type prism slip, $\vec{b} = \frac{1}{3}\left\langle 11\bar{2}0 \right\rangle$, gliding on prism $\left\{1\bar{1}00\right\}$ planes, and the twins were identified as $\left\{1\bar{1}02\right\}$ type.

2.4 Summary of the Results for Titanium Grade 7

The three-part study that included the analysis of the creep curves, the characterization of the microstructure of the as-received plate material, and the transmission electron microscopy investigation of the creep deformed specimens, provided important information in understanding the effect of microstructure, temperature, and stress level on the creep behavior of Titanium Grade 7.

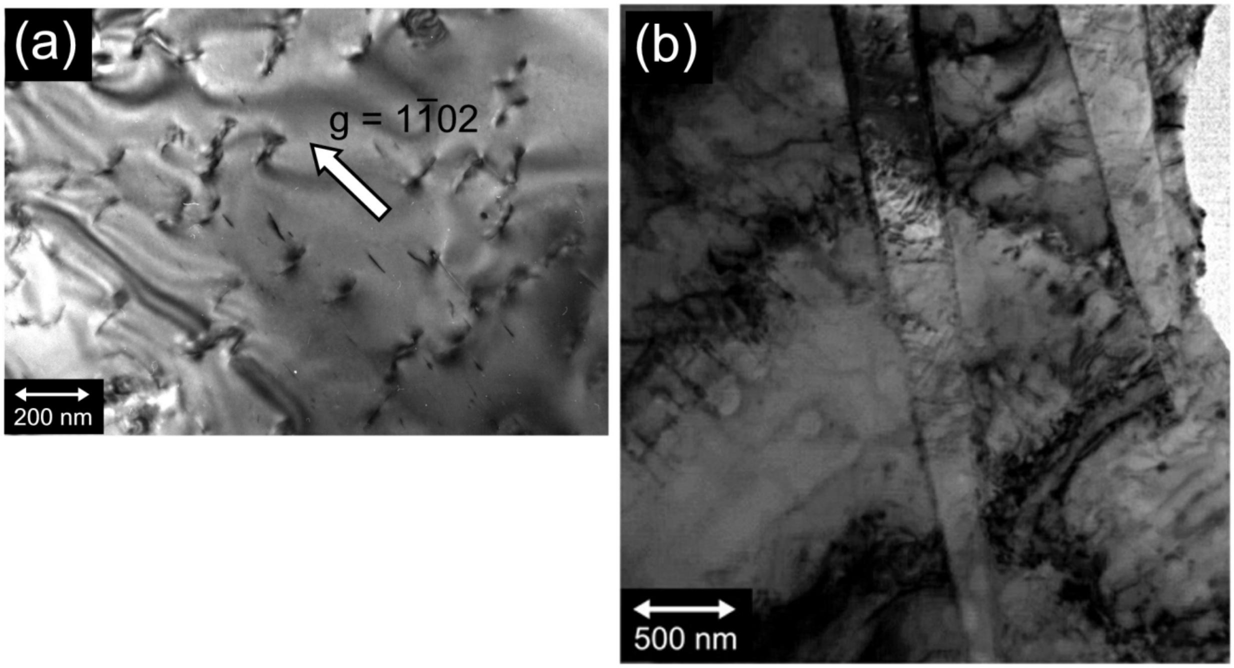


Figure 2-25. Bright-Field Transmission Electron Micrographs of Titanium Grade 7 Creep Tested at 25 °C [77 °F] at 85 Percent Yield Stress: (a) Array of *a*-Type Prism Dislocations and (b) Two Parallel $\left\{1\bar{1}02\right\}$ Type Twin

The deformation behavior is very sensitive to material grain size and texture. In particular, under the same loading conditions, the extent of twinning, and hence creep strain, increases as the grain size of a material increases (Meyers, et al., 2001; Aiyangar, et al., 2005). Therefore, the microstructure of the as-received plate material was examined. It was determined that the material has equiaxed grains with an average grain size of approximately 55 μm [0.0022 in].

Based on the results of the creep tests at various stress levels at 150 °C [302 °F], it was found that a logarithmic form equation [Eq. (2-2)] best describes the creep data and the creep strain increases exponentially with stress level. The threshold stress for creep strain at this temperature was found to be approximately 32.3 percent yield stress. This value has not been previously reported for Titanium Grade 7. The transmission electron microscopy investigation of the specimens tested at the various stress levels revealed that while slip is the predominant deformation mechanism at stresses less than the yield stress, at 100 percent yield stress, twinning is an active deformation mechanism. Hence, the material exhibits much more creep strain at 100 percent yield stress than at the lower stress levels. It is likely that for a larger-grained alloy, twinning would be an active deformation mechanism at the lower stress levels and the extent of creep would be greater.

For the specimens tested at various temperatures and at a constant stress level of 85 percent yield stress, unusual behavior was observed—the creep strain decreased as the temperature increased. Analysis of the tensile curves and the results of the transmission electron microscopy investigation, however, provided an explanation for this phenomenon. In the initial stages of creep, the strain rate for the elevated temperatures {100 and 150 °C [212 and 302 °F]} is greater than that of the lower temperatures {25 °C and 50 °C [77 and 122 °F]}. This is the strain region where slip will be the predominant deformation mechanism and more thermal energy at the elevated temperatures makes slip easier.

However, once a particular strain level is reached {approximately 1.25 percent for the specimen tested at 150 °C [302 °F] and approximately 2.25 percent for the specimen tested at 100 °C [212 °F]}, the strain rate decreases significantly and becomes less than that of the specimens tested at the lower temperatures. Transmission electron microscopy revealed that for the temperatures of 100 °C and 150 °C [212 and 302 °F], slip is the only active deformation mechanism, while at the temperatures of 25 °C and 50 °C [77 and 122 °F], slip and twinning are active deformation mechanisms. It is known that as temperature increases, slip is an easier deformation mechanism and thus twinning is less predominant (Song and Gray, 1995a). Therefore, for the specimens tested at the elevated temperatures, slip is the only predominant deformation mechanism. Furthermore, the load drop after yielding signifies that the applied stress level of 85 percent yield stress is significantly lower than the peak strain before the 0.2 percent yield stress is calculated. Hence, the local stress required to initiate twinning may not be reached.

For the specimens tested at the lower temperatures, deformation can continue by twinning in addition to slip and thus the creep deformation proceeds for long periods of time, resulting in higher creep strains at lower temperatures. The change in the predominant deformation mechanism (from slip to slip and twinning) with increasing strain at the lower temperatures is evidenced from the slight increase in activation energy for creep at 85 percent yield stress.

These findings show that it is critical to consider the microstructure and deformation mechanisms, as observed by optical and transmission electron microscopy, when interpreting the data from the creep tests at various temperatures and stress levels to fully understand the creep behavior of the alloy.