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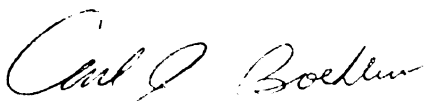
THE EFFECT OF PROCESSING ON THE PHYSICAL AND
MECHANICAL METALLURGY OF BORON-MODIFIED $\alpha+\beta$
TITANIUM ALLOYS

presented by

WEI CHEN

has been accepted towards fulfillment
of the requirements for the

Ph.D. degree in Materials Science and Engineering



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**THE EFFECT OF PROCESSING ON THE PHYSICAL AND
MECHANICAL METALLURGY OF BORON-MODIFIED $\alpha+\beta$ TITANIUM
ALLOYS**

By

Wei Chen

A DISSERTATION

**Submitted to
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ABSTRACT

THE EFFECT OF PROCESSING ON THE PHYSICAL AND MECHANICAL METALLURGY OF BORON-MODIFIED $\alpha+\beta$ TITANIUM ALLOYS

By

Wei Chen

This dissertation comprises a systematic study of boron (B) addition and processing on the microstructure, tensile behavior, 455°C fatigue behavior, and elevated-temperature creep behavior of two $\alpha+\beta$ alloys systems, namely Ti-6Al-4V (wt.%) (Ti-64) and Ti-6Al-2Sn-4Zr-2Mo-0.1Si (wt.%) (Ti-6242S). As-cast Ti-64-xB (x=0, 0.1, and 1 wt.%) and Ti-6242S-xB (x=0, 0.1, 0.4, and 1 wt.%) alloys were evaluated. For the Ti-64-1B alloy, the processing effects (including cast, cast-and-extruded, powder metallurgy (PM) rolled, and PM extruded) were investigated. For the as-cast processing condition, B-additions did not degrade the 455°C fatigue resistance of Ti-64 and Ti-6242S alloys, and the B content which resulted in the greatest average fatigue life and elongation-to-failure (ϵ_f) was 0.1 wt.% in both alloy systems. The creep resistance of the as-cast alloys was significantly improved with increased B concentration in the as-cast Ti-64-xB alloy system due to the load sharing effect of the strong and stiff TiB phase. However B-additions did not result in as significant of an improvement in the creep resistance of the Ti-6242S alloy system. The cast-and-extruded Ti-64-xB alloys exhibited significantly higher 455°C yield strengths, ultimate tensile strengths, and fatigue strengths than the as-cast Ti-64-xB alloys due to their smaller grain size, a smaller α -colony size, finer α -lath width, and strongly textured α -phase. For the same nominal B contents, the cast-and-

extruded alloys also exhibited significantly greater creep resistance and tensile strength than the as-cast alloys. The fatigue resistance of the PM processed Ti-64-1B alloys were superior to the as-cast and cast-and-extruded Ti-64-1B alloys due to the equiaxed α morphology in these alloys. The PM extruded Ti-64-1B exhibited a fine equiaxed α -phase morphology and strong α -texture, enabling it to exhibit even longer fatigue life than that of the PM rolled Ti-64-1B alloy. However, the creep resistance of the PM processed Ti-64-1B was worse than the cast-and-extruded Ti-64-1B alloy since creep property is favored by lenticular α morphology instead of equiaxed α morphology. Creep-fatigue interactions were also evaluated for the 455°C fatigue experiments. The large amount of dislocations generated by the fatigue process may have facilitated the creep deformation process and increased the strain rate. It is possible that the defects induced by fatigue (such as micro-voids) also increased the creep rate. The strong α -phase texture in the cast-and-extruded Ti-64-xB alloys and the PM extruded Ti-64-1B alloy resulted in the anisotropy of tensile strength. The YS and UTS for these alloys in the transverse direction were lower than in the longitudinal (extrusion) direction. As the α -phase texture became weaker with higher B content in the cast-and-extruded Ti-64-xB alloys, the tensile anisotropy became weaker. However, the tensile strength in the transverse direction was still much higher than that of the as-cast Ti-64 alloy. Overall, this dissertation work has shown that B-modified Ti-64 alloys show promise for fatigue-driven and creep-driven applications and have the potential to replace Ti-64 for both RT and elevated-temperature structural applications.

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Dedication

This dissertation is dedicated to my parents, Gang and Ming, who have been teaching, guiding and supporting my life throughout these years.

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CHAPTER 1

PROBLEM FORMULATION AND RESEARCH OBJECTIVES

A. Rationale

Titanium (Ti) and Ti alloys have been widely used in the aerospace industry due to their high specific strength and corrosion resistance. The aerospace industry has developed a series of chemical and thermomechanical processing routes to tailor the Ti alloys to arrive at desirable combinations of density, strength, fatigue and creep resistance. They have been used as aircraft fuselages, wings, missile bodies etc. due to the high payoff for weight reduction in the aerospace industry. Weight reduction is even more important in gas turbine engines than airframes especially for rotating components because of the centrifugal forces and the cascading effects. Currently, Ti alloys compose about one third the structural weight of modern turbine engines. Figure 1.1 illustrates the metals commonly used in a turbo fan engine.

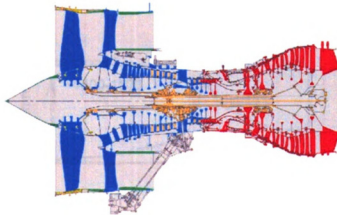


Figure 1.1. Metals used in a typical turbo fan engine. Ti alloys are in blue, nickel-based superalloys are in red, and steels are in yellow. [Trent 800 Engine, Rolls-Royce Plc.]

The working temperature of the turbine blades in gas turbine engines ranges from 650°C to 1000°C and the temperature profile as well as the pressure profile is shown in Figure 1.2. The longitudinal stresses in the turbine blades are about 140 MPa but the blade root experiences high tensile stress (280-560 MPa) [Reed-Hill and Abbaschian (1994)]. The working conditions require the turbine engine materials to not only withstand high temperature but also possess sufficient fatigue and creep resistance.

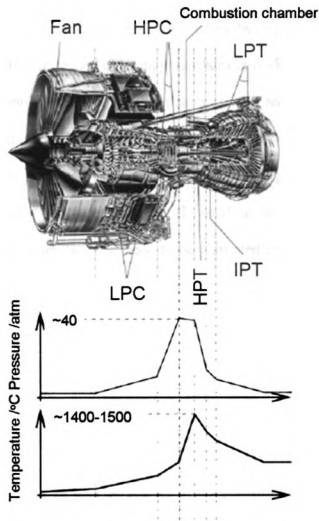


Figure 1.2. Rolls-Royce Trent 800 Jet Engine displaying the pressure and temperature profiles along the engine. (LPC-low pressure compressor, HPC-high pressure compressor, HPT-high pressure turbine, IPT-intermediate pressure turbine, and LPT-low pressure turbine.)

Figure 1.3 is a diagram of a twin-spool compressor in which the low-pressure compressor is typically made of Ti-64 alloy and the high-pressure compressor is typically made of Ti-6242S alloy or IMI 834 (Ti-5.8Al-4Sn-3.5Zr-0.7Nb-0.5Mo-0.35Si-0.06C) which can withstand higher temperature than the Ti-64 alloy. With the development of modern jet engines, the industry demands higher engine working temperatures in order to enhance the fuel efficiency and decrease pollution generation. For example, a single W501G power generating gas turbine (Siemens Westinghouse Power Corp.) would be expected to exhibit a 0.7% increase in efficiency when the engine working temperature is increased by 50°C. This increased efficiency translates into ~\$300,000/year savings from reduced fuel consumption and ~\$65,000/year savings from reduced emissions [Pratt and Whitney private communication (2003)]. Similar effects would also be expected in aerospace gas turbine engines. Therefore, to withstand higher temperature, these Ti alloys used as compressor blades and wheels need to be strengthened. The most important mechanical properties that need to be enhanced are the elevated-temperature fatigue and creep resistance.

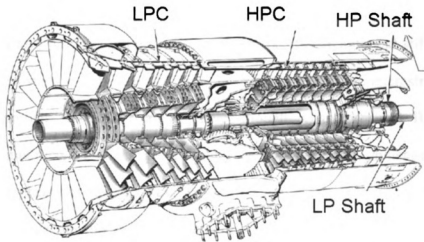


Figure 1.3. A diagram showing the structure of a twin-spool compressor. [The Jet Engine, Technical Publications Department, Rolls-Royce plc, Derby, England, 1996]

Conventional Ti alloys containing small additions of boron (B) have been gaining considerable interest in recent years due to their attractive mechanical properties including high room-temperature (RT) specific strength and Young's modulus (E) along with reasonable elongation-to-failure (ϵ_f) [Saito (2004), Ravi Chandran (2004), Tamirisakandala (2006), Sen (2007)]. The significant increase in strength and E values in B-modified Ti alloys arise from the strong and stiff TiB phase that precipitates *in-situ* during solidification [Panda (2006), Tamirisakandala (2005), Lu (2001)]. Due to the similarity of the thermal expansion coefficients ($8.6 \times 10^{-6}/^{\circ}\text{C}$ for Ti and $7.5 \times 10^{-6}/^{\circ}\text{C}$ for TiB [Ravi Chandran (2004)]), processing of such alloys is less challenging than for other ceramic-reinforced alloys. In addition, recent work has shown that the addition of trace amounts (~ 0.1 wt.%) of B to Ti-64, Ti-6242S, Ti-5553, and Timetal 215, significantly decreases the as-cast grain size [Tamirisakandala (2005), Tamirisakandala (2006), Cherukuri (2009)]. This drastic reduction in the as-cast grain size leads to significant benefits including increasing yield strength while reducing or avoiding time spent on expensive and energy-intensive thermomechanical processing. Small B additions do not result in significant density changes, but even so, the higher strength and stiffness of B-modified Ti alloys provides important increases in specific strength and stiffness. The aforementioned studies on B-modified Ti alloys show that dramatic microstructural and mechanical property alterations are possible through relatively small B additions. Although B-modified Ti alloys are currently used for various ambient-temperature commercial applications (e.g. exhaust valves of automotive engines, see Figure 1.4) [Saito (2004)], understanding the mechanisms of deformation and fracture is necessary to consider these materials for fracture-critical applications (e.g. aerospace) especially at

elevated temperature. This thesis focused exclusively on B-modified Ti-64 and Ti-6242S alloys since these alloys are the backbone of the Ti industry especially for aerospace applications (gas turbine engine blades and disks).

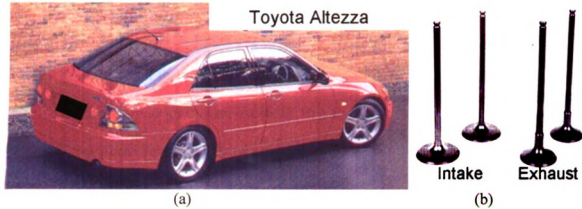


Figure 1.4. (a) The first family car (Toyota Altezza) to feature a Ti/TiB valve and (b) the appearance of the developed valves.

B. Research Objectives

The detailed aims of this study follow:

Microstructure

- (1) To understand the effect of B additions on the microstructure evolution of as-cast Ti-64 and Ti-6242S alloys.
- (2) To understand the effect of processing (extruding, rolling) on the microstructural features such as grain size, α -colony size, and α -phase morphology.
- (3) To evaluate the feasibility of using powder metallurgy for processing B-modified Ti-64 alloys and compare the microstructures of powder metallurgy processed and ingot metallurgy processed Ti-64-xB alloys.
- (4) To quantify the microstructural features of the studied alloys, such as grain size, phase volume fraction, α colony size, and lath width in order to obtain an understanding of the possible influence of microstructure on the mechanical properties.
- (5) To determine the texture of Ti-64-xB alloys under different processing conditions using Electron Backscatter Diffraction (EBSD) and X-ray Diffraction (XRD), in order to evaluate the effect of texture on the mechanical properties.

Tensile Behavior

- (1) To obtain the RT and elevated-temperature (400°C, 455°C, 510°C, 565°C) tensile properties of the studied alloys, to assist in the development of processing-microstructure-tensile relationships.

- (2) To obtain the RT and elevated-temperature tensile strength in the transverse direction of the extruded and rolled Ti-64-xB alloys in order to verify if the texture resulted in tensile anisotropy in the Ti-64-xB alloys.
- (3) To quantify the effect of α -phase texture and TiB-phase texture on the Young's modulus of extruded Ti-64-xB alloys.
- (4) To track the tensile deformation evolution of the TiB, α and β phases through *in-situ* tensile experiments inside an SEM.
- (5) To choose the stress levels for the fatigue and creep tests based on the tensile properties of the studied alloys.

Fatigue Behavior

- (1) To obtain the fatigue S-N data at 455°C and stress range of 250 – 700 MPa (R=0.1) for Ti-64-xB alloys, and 300 – 500 MPa (R=0.1) for Ti-6242S-xB alloys.
- (2) To evaluate the effect of B addition and processing on the fatigue behavior of Ti-64 and Ti-6242S alloys to assist in the development of processing-microstructure-fatigue relationships.
- (3) To characterize the fatigue deformation evolution and the fatigue crack growth during the fatigue experiments by performing *in-situ* fatigue tests in an SEM.
- (4) To evaluate the Young's modulus change during fatigue tests by plotting the stress-strain hysteresis at certain cycle numbers. This was used to correlate the stress-strain hysteresis with TiB-phase cracking.
- (5) To evaluate and compare the endurance limit (EL) for Ti-64-xB and Ti-6242S-xB and evaluate the ratio EL to UTS.

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Creep Behavior

- (1) To determine the creep strain dependency on temperature and stress over the temperature range of 400 – 455°C and stress range of 400 – 600MPa for the Ti-64-xB alloys, which underwent different processing conditions.
- (2) To determine creep strain dependency on temperature and stress over the temperature range of 455 – 565°C and stress range of 138 – 600 MPa of the as-cast Ti-6242S-xB alloys.
- (3) To obtain the steady-state creep rate at different testing conditions in order to compare these values to those found in literature for similar alloys. These values were used to calculate the creep stress exponents and apparent activation energies.
- (4) To characterize the creep deformation evolution by performing *in-situ* creep tests in an SEM.
- (5) To evaluate the dominant secondary creep mechanisms by evaluating the deformation behavior and the steady-state creep rate versus stress and temperature dependence.

Fatigue-Creep Interaction

- (1) To evaluate the contribution of the total fatigue strain that could be attributed to creep for the fatigue tested Ti-64-xB alloy samples.
- (2) To understand the fatigue-facilitated creep process during the elevated-temperature fatigue tests. This was done by comparing the creep strain rate during the fatigue tests to the creep strain rate during the conventional creep tests.

C. Expected Outcomes in This Dissertation

In order to setup the basis and relevance of this work, I would like to put forward some of the some of the expected outcomes. None of the following expected outcomes were evident in the literature at the time this dissertation work was initiated back in the fall of 2006 when I started graduate school at MSU:

- (1) B will increase the strength of Ti-64 alloy where increase B increases E value and UTS but decreases ϵ_f .
- (2) Comparing with as-cast Ti-64-xB alloys, wrought Ti-64-xB alloys would exhibit greater fatigue lives.
- (3) PM processed alloys exhibit greater fatigue lives than as-cast or cast-and-extruded Ti-64-xB alloys.
- (4) B-modified as-cast Ti-64 and Ti-6242S alloys would exhibit better fatigue resistance than the baseline Ti-64 and Ti-6242S alloys.

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CHAPTER 2

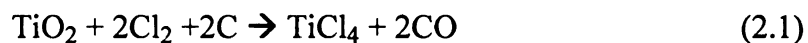
BACKGROUND AND LITERATURE REVIEW

A. Fundamentals of Ti and Ti-B Alloys

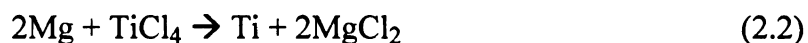
1. History of Ti Alloys

Titanium (Ti) ranks as the 9th most plentiful element and the 4th most abundant structural element in the earth's crust [Boyer (1994)]. The name "titanium" came from the Titans, children of Uranos and Gaia in Greek mythology. This name was given in 1795 by Martin H. Klaproth, who isolated Ti oxide from a mineral now known as "rutile" [Prior and Zambonini (1908)]. Actually, Ti oxide was discovered four years earlier by William Gregor, but he named it "mechanite". It took more than 100 years to isolate the metal Ti from ore, similar to the fate of the Titans who were held in captivity in the earth's crust by their father [West (1971)].

In the commercial production of Ti metal, rutile is first chlorinated in the presence of coke or another form of carbon:



Then the metal Ti is extracted from TiCl_4 ("tickle") and reduced to Ti sponge using Mg (Kroll process [named after Wilhelm J. Kroll]) or Na (Hunter process [named after Matthew A. Hunter]). Today, the Kroll process is the most widely used method for producing Ti. The basic reaction of Kroll process is



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The Ti sponge is then vacuum distilled, swept with an inert gas, or acid leached to reduce the remnant salt content [Froes (2004)].

Stimulated by the recognition of the aluminum (Al) additions for strengthening, the development of Ti alloys progressed rapidly after World War II and they became key materials for aircraft engines. In fact, today two-thirds of all the Ti metal produced is used in aircraft engines and frames [Boyer (1994)] due to its high specific strength and corrosion resistance. The weight percentage of Ti alloys in military aircraft fuselages can exceed 50%. For the SR-71 “Blackbird” reconnaissance aircraft, the weight percentage of Ti alloys in the fuselage was 95% [Peters (2003)]. Other industries, such as architecture, chemical processing, medicine, power generation, sports, marine and offshore, and transportation are currently witnessing an increased usage of Ti [Peters (2003)].

2. Metallurgy of Ti Alloys

Pure Ti is allotropic and has two solid crystalline states: a low-temperature α phase, which is hexagonally close packed (HCP), and a high-temperature β phase, which is body-centered cubic (BCC). For pure Ti, the transition temperature (β -transus temperature) at which this crystallographic phase transformation occurs is $882\pm 2^\circ\text{C}$ [Peters (2003)]. The allotropic transformation enables the formation of Ti alloys composed of α , β , and $\alpha+\beta$ microstructures. Different alloying elements can increase or decrease the β -transus temperature and thus stabilize either the α or β phases. Elements such as Al, O, N, C, B etc. are typical α stabilizers and V, Zr, Nb, Mo, Fe, etc. are typical β stabilizers [Boyer (1994)].

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The arrangement, volume fraction, sizes and individual properties of the α and β phases are the basis for the variety of properties exhibited by Ti alloys. Once the chemistry of the Ti alloys is selected, the modification of the mechanical properties is done by working to tailor the microstructure such as the size, morphology, and dispersion of the α and β phases. In general, α alloys exhibit better creep resistance than β alloys do since the diffusion rate in α -Ti is more than two orders of magnitude lower than that of the β -Ti [Peters (2003)]. In general, α alloys exhibit superior weldability but inferior forgeability compared with $\alpha+\beta$ and β alloys [Peters (2003)]. The major α alloy is the commercially pure α -Ti with different oxygen and iron contents [Boyer (1994)]. The commonly used β alloys are actually all metastable because stable β alloys do not exist as commercial products [Lutjering (2007)]. The strengths of β alloys can achieve values higher than 1400 MPa through heat treatment, and the complex microstructure of β alloys enables designers to optimize them for both high strength and high toughness [Peters (2003)]. Ti-6Al-2Sn-4Zr-6Mo (Ti-6246) and Ti-5Al-2Sn-2Zr-4Mo-4Cr (Ti-17) are typical commercial β alloys.

The $\alpha+\beta$ alloys support a mixture of α and β phases in equilibrium at RT and they usually contain both α and β stabilizers [Boyer (1994)]. The two-phase microstructure can provide well-balanced mechanical properties and the alloys generally exhibit good fabricability, high RT strength and moderate elevated-temperature strength [Boyer (1994)]. Among all the $\alpha+\beta$ alloys, the Ti-6Al-4V (Ti-64) is the alloy most prevalently used. It is the most widely used Ti alloy and accounts for more than 50% of all the Ti tonnage in the world [Boyer (1994)]. More than 80% of its usage is in aerospace industry [Boyer (1994)]. It can be readily welded, forged and machined into various forms such

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as sheet, plate, wire, and rod. Ti-64, along with another $\alpha+\beta$ alloy: Ti-6Al-2Sn-4Zr-2Mo-0.1Si (Ti-6242S), are the backbone of the Ti industry, especially for aerospace applications.

In addition, intermetallic Ti-Al alloys offer enhanced high-temperature mechanical properties compared with α , β , and $\alpha+\beta$ Ti alloys [Froes (1992), Froes (1994)]. They are also lighter as Al is much lighter than Ti and Al consist of up to 50 at.% for these intermetallic systems. These intermetallics have the potential to replace nickel-based superalloys commonly used in gas turbine engines (working temperature up to 900°C) based on their density-normalized properties. The two Ti-based intermetallics receiving the most attention have been the ordered HCP Ti_3Al (α_2) alloys and the face-centered tetragonal TiAl (γ) alloys. However, the lack of ductility of these structural intermetallics, due to the limited number of active slip systems, limits their application.

3. History of Ti-B alloys

The earliest work on Ti alloyed with B was conducted by Armour Research Foundation (IIT) in the mid 1950s and by Royal Aircraft Establishment in the late 1950s. B was found to be highly effective for increasing the stiffness of Ti alloys (0.5 wt.% of B addition has an effect equivalent to that of adding 6.5 wt% of Al).

However, the the Ti-B alloys were not commercialized until the 1980s. In the early 1980s, Dynamet Technologies, Inc. used a powder metallurgy process (blended powder approach with cold and hot isostatic pressing) to produce alloys with discontinuous reinforcement [Miracle (2005)]. TiC was used as the reinforcement and TiB and TiB_2 were also considered. The reinforced alloys were made into commercials

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products such as skate blades, softball bats, and automotive valves [Abkowitz (2005)]. Dynamet Technologies, Inc. continued its research and development on Ti-B alloys. The mechanical properties of the recent Ti-B alloy using the cold and hot isostatic pressing method have achieved 896 MPa for YS, 965 MPa for UTS, and 14% for ϵ_f [Abkowitz (2005)].

In the mid 1990s, NASA collaborated with Boeing on the High Speed Civil Transport (HSCT) project. The goal of this project was to improve the aircraft structural efficiency by developing alloys with good durability, ductility and damage tolerance [Schwendiman (2007)]. Ti-B alloys were one of the candidate alloys. As a part of the HSCT project, several Ti-B alloys (and baseline Ti alloys without B additions) were cast into 3.0 inch diameter billets and extruded to 0.75 inch diameter for evaluating their mechanical properties. The B-modified Ti alloys showed a greater than 10% increase in Young's modulus and strength along with a decent ϵ_f value (see Table. 2.1). Figure 2.1 shows two extrusions of Ti-6Al-2Fe-0.1Si-0.6B (Ti-62S-0.6B) alloys developed by the HSCT project. However, the work on Ti-B alloys was discontinued due to the poor fatigue initiation characteristics, and poor transverse mechanical properties of these extruded alloys [Government contract No. NASI-20220].

Table 2.1. Mechanical Properties of Ti-B Alloys (longitudinal direction) from HSCT Project [Government contract No. NASI-20220: High Speed Research-Airframe Technology Report]

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64 (baseline)	Annealed	110	924	1000	10
Ti-64-0.5B	Annealed	124	986	1076	10
Ti-64-0.5B	STA	126	1186	1282	7
Ti-21S (baseline)	STA	101	1179	1234	7
Ti-21S-0.5B	STA	112	1220	1276	7
Ti-6-22-22 (baseline)	Annealed	116	1007	1096	10
Ti-6-22-22-0.5B	Annealed	126	1087	1219	8.1
Ti-6-22-22-0.8B	Annealed	129	1163	1271	6.2
Ti-62S-0.5B	Annealed	135	1075	1128	6.1
Ti-62S-0.8B	Annealed	136	1071	1129	7.2

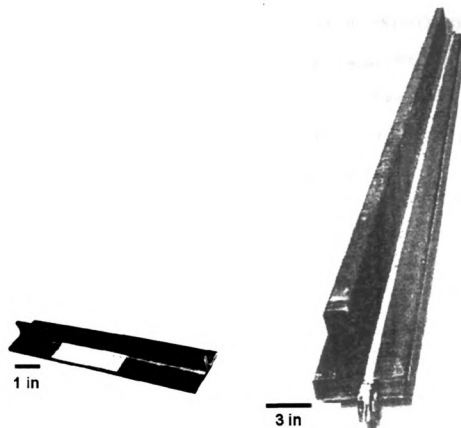


Figure 2.1. High modulus Ti-62S-0.6B extrusions manufactured during the HSCT project. [Government contract No. NASI-20220: High Speed Research-Airframe Technology report]

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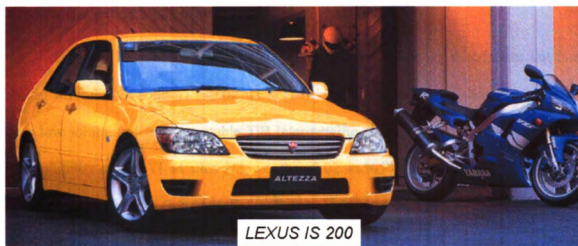
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In the late 1990s, Toyota Motor Corp. succeeded in being the first mass production automobile manufacturer to apply Ti-B alloys in engine components (see Figure 2.2a). A PM process was used to manufacture the intake (Ti-64/TiB alloy) and exhaust valves (Ti-6Al-4Sn-4Zr-1Nb-1Mo-0.2Si/TiB alloy), see Figure 2.2b. During the development of the discontinuously reinforced Ti alloys, TiB was found to be the best reinforcement and it outperformed all existing reinforcements such as SiC, TiC, TiB₂, B₄C, TiN, SiN₄, and Al₃O₄ [Saito (2005)]. Comparing with the steel valves, Ti-B valves are 40% lighter, resulting in a 16% lighter spring valve weight, a 30% noise reduction, a 20% camshaft driving torque reduction, and an increase in maximum revolutions of 700 rpm culminating in high performance and low fuel consumption [Saito (2004)]. The first car to use these valves was the Toyota Altezza (Figure 2.2a) in 1998. The valves were later adapted and applied to Yamaha motorcycle (Figure 2.2a) [Saito (2005)]. The Ti-B alloys were also used in the valves of the Nissan's Infiniti V-8 motor and the power train connecting rods in the Acura NSX V-6 engine [Peters (2003)].



(a)



(b)

Figure 2.2. (a) Toyota Altezza vehicle (left) and Yamaha motorcycle (right) use Ti-B alloys for engine valves, and (b) the intake (Ti-64/TiB alloy) and exhaust valves (Ti-6Al-4Sn-4Zr-1Nb-1Mo-0.2Si/TiB alloy) used in Toyota Altezza vehicles. [Saito (2005)]

4. Metallurgy of Ti-B alloys

Figure 2.3 shows the Ti-B binary phase diagram. B is an α stabilizer but it is almost insoluble in solid Ti (<0.02 wt.% [Palty (1954)]) under equilibrium conditions. This low solubility leads to a stable TiB intermetallic phase through a eutectic reaction. The solubility of B in Ti-64 alloy is lower than 0.02 wt.% [Tamirisakandala (2005)]. The eutectic composition is 2.2 wt.% B for Ti (see Figure 2.3) and is estimated to be 1.56 wt.% for Ti-64 [Ivasishin (2005)]. In addition, B addition was also reported to increase the β transus of Ti-64 [Tamirisakandala (2005), Soboyejo (1994)]. An increase of β transus by 60°C was observed in Ti-64-1.7B alloy and this increase was attributed to the equilibrium as well as the supersaturated B in the solid solution [Tamirisakandala (2005)].

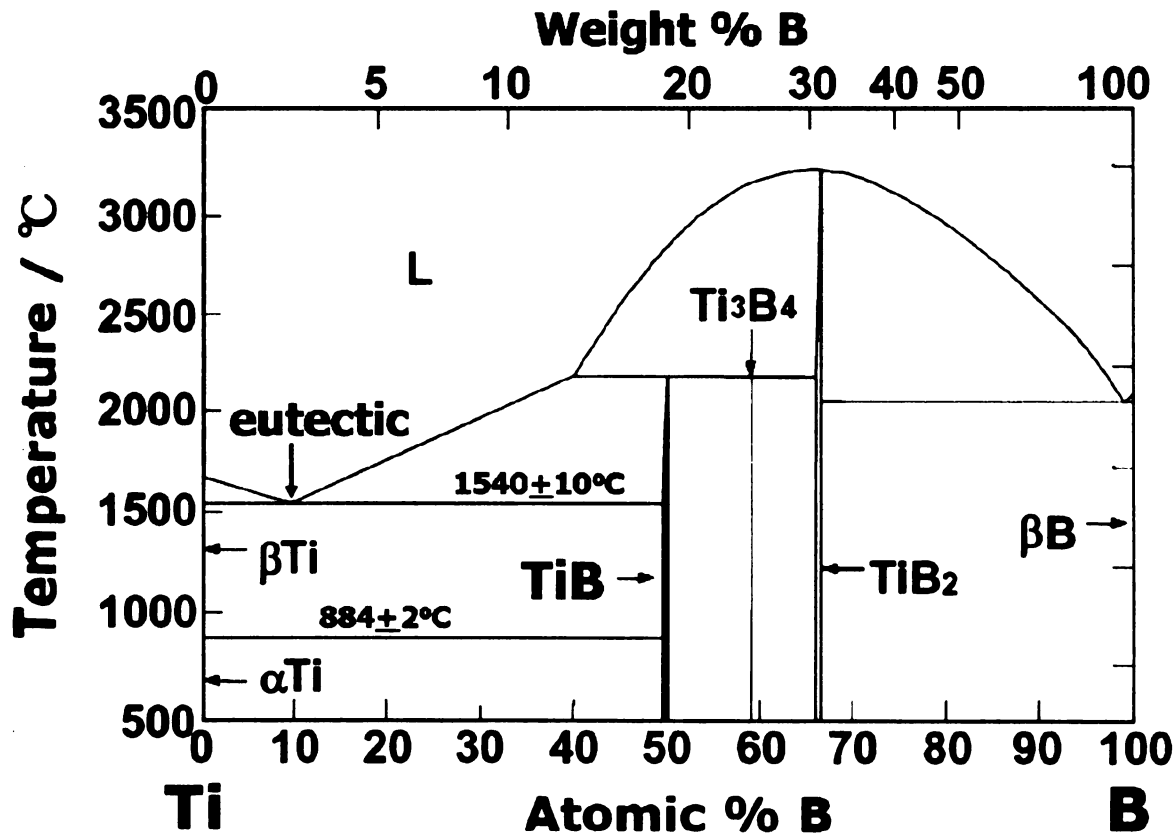


Figure 2.3. Ti-B binary phase diagram [Murray (1992)]

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TiB can be formed *in-situ* by reaction of Ti and B through:



or by Ti and TiB₂ through:



Extensive studies on the kinetics and thermodynamics of the Ti-B alloy system have shown that TiB is only stable in a Ti matrix. [Ma (2004), Zhang (2003)]. Thus it is impossible to synthesize TiB single crystals for characterization and no experimental mechanical properties have been reported for TiB. The TiB phase is more stable than TiB₂ if and only if excess Ti is present. It is noted that it would be difficult to extract the TiB phase from the Ti matrix, and even if one was able to do this, it would be difficult to mechanically test the extremely small TiB phase.

The TiB phase has an orthorhombic (B27) crystal structure with $a=6.12 \text{ \AA}$, $b=3.06 \text{ \AA}$, and $c=4.56 \text{ \AA}$ [Decker (1954)]. The arrangement of atoms is illustrated in Figure 2.4. B atoms form a zigzag chain along the [010] direction with each B atom lying at the center of a trigonal prism of 6 Ti atoms [Panda (2006)]. {010} planes are the closest packed planes of TiB and [010] is its growth direction [Yang (1995), Tian (2008)], which leads to the needle-like morphology of TiB that develops due to the anisotropic characteristic of the B diffusion [Sahay (1999)]. The typical TiB phase in the Ti matrix is whisker-like single crystals with a roughly hexagonal cross-section. A metastable form of TiB was also reported, which has a B_f structure of fine needles with a high density of stacking faults [Kooi (2003), Hyman (1991)].

Comparing with other common particle reinforcements such as TiC, SiC, B₄C and Al₂O₃, TiB has the best thermodynamic compatibility and thermal-expansion

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compatibility with Ti. The coefficient of linear thermal-expansion of TiB is approximately $7.5 \times 10^{-6}/^{\circ}\text{C}$, which is quite close to the corresponding value for Ti ($8.6 \times 10^{-6}/^{\circ}\text{C}$) [Ravi Chandran (2004)]. This similarity of the thermal expansion coefficients makes processing of Ti-B alloys less challenging than for other ceramic-reinforced alloys [Saito (2004), Ravi Chandran (2004)].

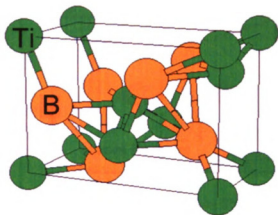


Figure 2.4. Crystal structure of TiB (orthorhombic, B27).

5. Ti-B Alloy Processing and Microstructure

A variety of processing methods have proven successful in manufacturing Ti-B alloys [Zhang (2003), Peng (1996), Kooi (2003), Geng (2004), Tamirisakandala (2004), Kawabata (2004), Saito (2004)]. The Ti-B engine valves used in Toyota Altezza Vehicles were manufactured through a blended elemental PM process, see Figure 2.5. The process starts with Ti, TiB_2 and Al-25Sn-25Zr-6Nb-6Mo-1.2Si wt.% powders and produces the Ti-B billets through powder mixing, compacting, and vacuum sintering. Then the billets are made into valves through a series of thermomechanical processing [Saito (2005)].

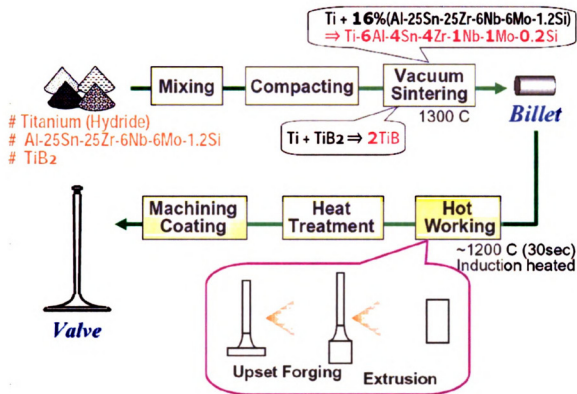


Figure 2.5. Blended elemental PM process flow chart for manufacturing Ti-B engine valves. [Saito (2005)]

The blended elemental PM process developed by Toyota created a cost-effective way of manufacturing Ti-B engine valves and has the most promise for low cost manufacturing of other Ti-B parts in automotive industry. However, this process is insufficient for meeting the rigorous needs for the aerospace industry where the standards for material strength, fatigue and creep resistance are much higher.

The Air Force Research Laboratory (AFRL), Wright-Patterson, OH has been developing Ti-B alloys for military applications in recent years. The Materials and Manufacturing Directorate of AFRL, in collaboration with their industrial and academic partners, has been focusing on the processing methods of Ti-B alloys in order to assist development of the processing-microstructure-property relationships for the Ti-B alloy

system. The researchers at AFRL and FMW Composites, Inc. (Bridgeport, WV) have explored a wide range of processing methods for producing Ti-B alloys [Tamirisakandala (2005)]. The processing routes they have been investigating are cast, cast and wrought, blended elemental PM, and pre-alloyed PM processes, see Figure 2.6. The blended elemental (BE) PM process, similar to the process used by Toyota for manufacturing Ti-B alloy engine valves, can obtain higher volume fractions of TiB without the formation of primary TiB because the entire process is performed in solid state [Tamirisakandala, (2004)]. However, the BE PM process results in a coarse microstructure due to long thermomechanical processing time at high temperature to convert the B source (usually B or TiB₂) to TiB. Comparing with the BE PM process, the pre-alloyed (PA) PM process is able to produce finer scale microstructural features due to fast solidification from Ti-B alloy melt, but it is limited to the hypoeutectic compositions (B<1.6 wt.%) to avoid the formation of primary TiB [Tamirisakandala (2004)].

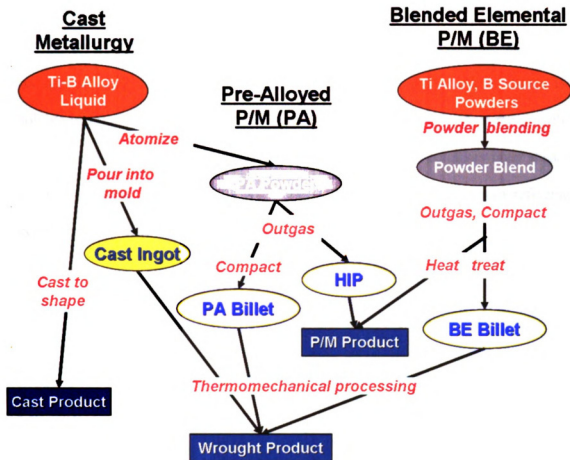


Figure 2.6. Typical processing methods for Ti-B alloys. [Miracle (2005)]

The cast metallurgy process of Ti-B alloys is also an important research emphasis. Recent work at AFRL has shown that the addition of small amounts of B can decrease the as-cast grain size of Ti alloys significantly [Tamirisakandala (2005), Sen (2007), Cherukuri (2009)]. This drastic reduction in the as-cast grain size can be beneficial as it reduces or avoids time spent on expensive and energy-intensive thermomechanical processing. Actually, the grain refinement effect by B addition was first reported by Zhu *et al.* in 2003 [Zhu (2003)], but the mechanism was not explained in detail until later. Tamirisakandala *et al.* [Tamirisakandala (2005)] studied the grain refinement effect of B in as-cast Ti-64 and Ti-6242S alloys, and 0.1 wt.% B was found to decrease the as-cast

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grain size by almost an order of magnitude, see Figure 2.7. They suggested that B additions enhance the nucleation rate of prior- β grain by providing additional driving force and slowing the growth rate by affecting the liquid/solid interface. Since B is insoluble in solid Ti, the B rejected at the solid/liquid interface can restrict the growth of existing nuclei and the rejected B will accumulate in an enriched boundary layer ahead of the interface [Kurz (1984)]. This was suggested to be the possible grain refinement mechanism in Ti-B as-cast alloys and in turn this causes the TiB to accumulate at the prior- β grain boundaries.

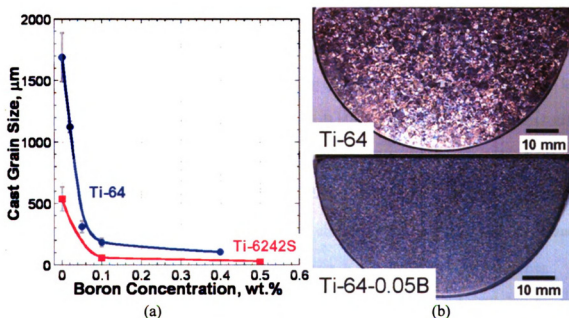


Figure 2.7. Grain refinement effect by B addition in Ti-64 and Ti-6242S alloys. (a) As-cast grain size versus B concentration plot, and (b) digital photographs showing the as-cast grain size difference between Ti-64 and Ti-64-0.05B alloys. [Tamirisakandala (2005)]

Banerjee *et al.* [Banerjee (2004), Banerjee (2005), Genc (2006)], of the Center of the Accelerated Maturation of Materials (Camm) at Ohio State University (OSU), have conducted extensive research on producing Ti-B alloys using the laser-engineered net-

shaping (LENS) process. In this process, successive layers of powder metal feedstock are melted using a high-powered laser and the final Ti-B alloys possess refined and homogeneous microstructures.

6. Tensile Properties of Ti-B Alloys

The attractive mechanical properties of Ti-B alloys have been the primary reason why such alloys have been gaining considerable interest in recent years. Their high room-temperature (RT) specific strength and Young's modulus (E) along with reasonable elongation-to-failure (ϵ_f) have been reported by many research groups [Soboyejo (1994), Saito (2004), Ravi Chandran (2004), Schuh (2001), Panda (2006), Tamirisakandala (2005), Bilous (2005), Gorsse (2003), Soboyejo (2004), Boehlert (2006)]. The significant increase in strength and E values in Ti-B alloys arises from the strong and stiff TiB phase ($E \sim 371\text{--}485$ GPa) that precipitates *in-situ* during solidification or solid state transformation [Tamirisakandala (2005), Panda (2006), Gorsse (2003)]. Small B additions do not result in significant density changes, thus the higher strength and stiffness of Ti-B alloys provides important increases in specific strength and stiffness.

The typical RT Young's modulus, yield strength (YS) and ultimate tensile strength (UTS) for as-cast Ti-64-xB alloys are listed in Table 2.2. For the as-cast Ti-64-1.55B, only an E value was reported because the sample fractured below the YS [Ivasishin (2008)], indicating a brittle feature of this alloy. The RT ϵ_f value of as-cast Ti-64-xB alloys with respect to B concentration has been studied and the result is shown in Figure 2.8 [Miracle (2005)], which indicates that more than 0.4 wt.% of B resulted in poor ductility of as-cast Ti-64-xB alloys.

Table 2.2. RT Tensile Properties of As-cast Ti-64-xB Alloys

Alloy	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64*	107	834	900	6
Ti-64-0.05B*	110	879	944	8.5
Ti-64-0.1B*	116	900	966	7.3
Ti-64-0.4B*	126	913	973	5.2
Ti-64-1.55B**	134	N/A	N/A	N/A

N/A: not available

*[Sen (2007)], ** [Ivasishin (2008)]

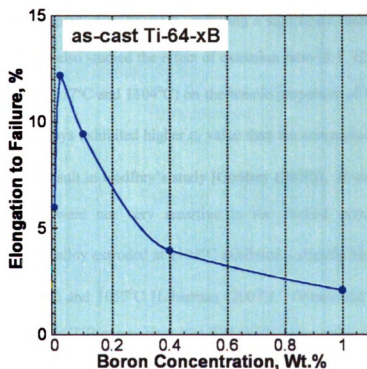
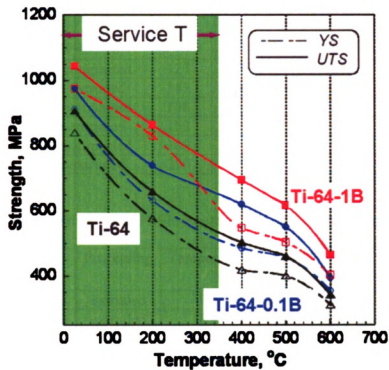


Figure 2.8. RT ϵ_f values of as-cast Ti-64-xB alloys versus B concentration. [Miracle (2005)]

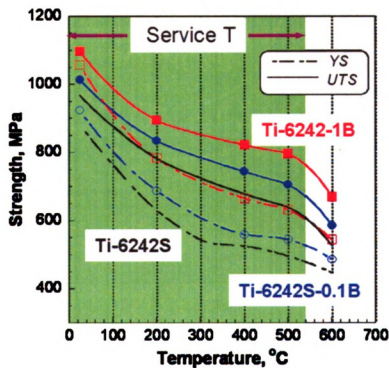
The B-modified Ti-6242S alloys were not studied as extensively as the B-modified Ti-64 alloys. The preliminary research performed at AFRL indicates that similar to the Ti-64-xB alloys, B addition also has the strengthening effect on the Ti-6242S alloy [Miracle (2005)]. The strengthening effect due to B addition is maintained

at elevated temperatures for as-cast Ti-64-xB and Ti-6242S-xB alloys [Miracle (2005)], offering the potential for the elevated-temperature applications of these alloys, see Figure 2.9.

The tensile properties of Ti-B alloys produced by the PM process were also investigated by several researchers. Liberman systematically studied tensile properties of PM processed Ti-64-xB alloys produced by PA and BE processes [Liberman (2007)]. The results are shown in Table 2.3. For the Ti-64-2.9B alloy in that study, the strong and stiff TiB whiskers occupied over 20 vol.%, resulting a significant increase of YS, UTS and E value. Liberman also studied the effect of extrusion ratio (8:1, 12:1, and 16:1) and temperature (1010°C, 1057°C and 1104°C) on the tensile properties of PA PM Ti-64-1B alloy. The extruded alloys exhibited higher ϵ_f value than the compacted alloys, which is in consistent with the result in Godfrey's study [Godfrey (2000)]. It was also found that the tensile properties were not very sensitive to the studied extrusion conditions, However, the Ti-64-1B alloy extruded at 1104°C exhibited a slightly higher ϵ_f value than that extruded at 1010°C and 1057°C [Liberman (2007)]. Gorsse and Miracle [Gorsse (2003)] studied the Ti-64/TiB composites (the TiB vol.% were up to 40% thus they are considered composites instead of alloys) produced by BE PM process and reported their tensile properties at RT and 300°C, see Table 2.4.



(a)



(b)

Figure 2.9. Elevated-temperature tensile properties of B-modified (a) as-cast Ti-64, and (b) as-cast Ti-6242S alloys. [Miracle(2005)]

Table 2.3. RT Tensile Properties of PM Processed Ti-64-xB Alloys [Liberman (2007)]

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64-1B	PA compacted	127	927	1038	6.2
Ti-64-1B	PA extruded	143	1078	1214	13.3
Ti-64-1.6B	PA compacted	138	1232	1347	2.4
Ti-64-1.6B	PA extruded	152	1350	1420	4.0
Ti-64-2.9B	BE compacted	160	1107	1119	1.5
Ti-64-2.9B	BE extruded	200	1189	1331	3.0

Table 2.4. Tensile Properties of BE PM Processed Ti-64/TiB Composites [Gorsse (2003)]

Materials	Processing	Temperature	E GPa	YS MPa	UTS MPa	ϵ_f %
Ti-64/20% TiB	extruded	RT	170	1181	1215	0.5
Ti-64/40% TiB	extruded	RT	210	-	864	0
Ti-64/20% TiB	compacted	RT	145	-	1018	0.1
Ti-64/20% TiB	extruded	300°C	160	823	910	1.9
Ti-64/40% TiB	extruded	300°C	207	871	871	0.2
Ti-64/20% TiB	compacted	300°C	140	674	748	0.9

B. Fatigue Background

1. Fundamentals of Fatigue

When a cyclic load is applied to a material, fatigue typically occurs. A fatigue nucleus can initiate on a microscopic scale, followed by crack growth to a macroscopic scale, and final failure in the last cycle of the fatigue life. The fatigue life until failure is usually divided into two stages: crack initiation period (including crack nucleation and crack growth of microstructurally small cracks), and crack growth period (including crack growth into the bulk material) [Schijve (2009)]. The different stages of fatigue life and relevant factors are summarized in Figure 2.10 [Schijve (2009)]: the stress concentration factor (K_t) is the most important parameter for predictions on crack initiation; the stress intensity factor (K) is used for predictions on crack growth; for the last cycle of the fatigue life, at which the material breaks, the fracture toughness (K_{IC}) is the most important factor to be considered.

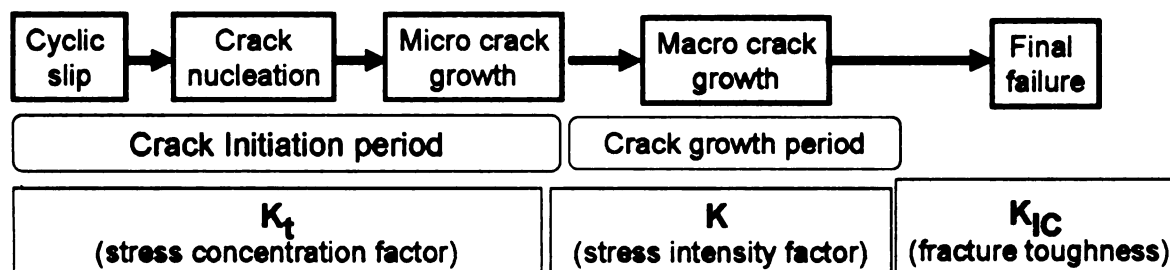


Figure 2.10. Different stages of the fatigue life and relevant factors [Schijve (2009)]

The lower restraint on cyclic slip at the material surface is a favorable condition for crack initiation. Crack initiation is very sensitive to surface conditions such as surface roughness, fretting, corrosion pits, etc. Thus fatigue is known as a material

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amplitudes, the crack initiation life continuously increases. Therefore, low-cycle fatigue (LCF) is dominated by fatigue crack propagation while high-cycle fatigue (HCF) is dominated by fatigue crack initiation [Peters (2003)].

The fatigue properties of Ti alloys highly depend on the microstructure. The most influential microstructural features include the grain size (or phase dimension and morphology), the age hardening condition, the degree of work hardening, the elastic constants, and the crystallographic texture [Peters (2003)].

For $\alpha+\beta$ Ti alloys, such as Ti-64 and Ti-6242S, the α phase morphology is critical. It is well established that low-cycle fatigue (LCF) behavior is favored by equiaxed α morphology [Froes, (2004)] and smaller grain size (equiaxed α) results in better fatigue properties [Lutjering (2007)]. Reducing the α grain size from 12 to 2 μm in equiaxed microstructures increased the fatigue strength of a Ti-64 alloy from 560 to 720 MPa [Peters (1983)]. For a lenticular microstructure, smaller α lath is beneficial for fatigue properties. It has been reported that reducing the α lath width from 10 to 0.5 μm raises the fatigue strength from 480 to 675 MPa for a Ti-64 alloy [Peters (1983), Trojahn (1980)].

Different processing methods will create Ti alloys with different microstructures. Hertzberg [Hertzberg (1996)] summarized the processing effect on the RT fatigue properties of Ti-64 alloy, see Figure 2.12. The wrought alloy exhibited greater fatigue lives, followed by the HIPped pre-alloyed powder and the cast alloys. It is not yet clear if the Ti-B alloys follow the same trend as Ti alloys but this will be evaluated in this dissertation.

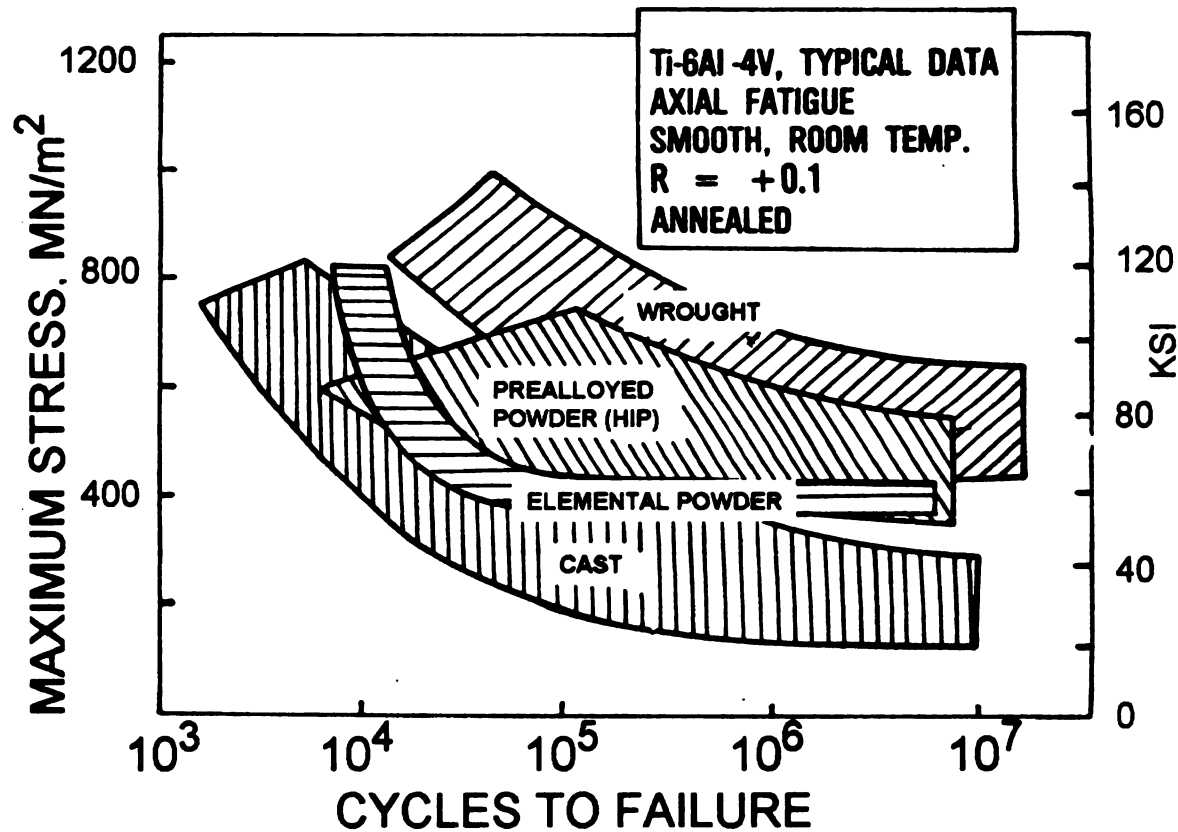


Figure 2.12. Typical RT fatigue data for Ti-64 alloy with different processing [Hertzberg (1996)]

3. Fatigue of Ti-B Alloys

As early as 1995, Soboyejo *et al.* [Soboyejo (1995)] reported that the BE PM extruded Ti-64-0.5B, Ti-6Al-0.5B and Ti-7.5Al-4V-0.5B alloys have superior fatigue crack growth resistance than mill annealed Ti-64 at RT. The crack growth rate of Ti-6Al-0.5B alloy was about five times lower than that of mill annealed Ti-64 alloy. They suggested that the fatigue crack growth resistance in the three B-modified Ti alloys was enhanced by the TiB whiskers. In 2004, Soboyejo *et al.* [Soboyejo (2004)], reported the RT crack nucleation and growth in cast-and-extruded Ti-64-0.5B alloys. In their study, cracks initiated at approximately 20% of the total fatigue life, indicating that the fatigue behavior was crack growth dominated. Crack blunting by the β phase in the Ti-64

matrix was also observed. They found that fatigue crack growth rates in the Paris regime in the Ti-64-0.5B alloy are comparable to those of the baseline Ti-64 alloy. However, in the low ΔK regime, the Ti-64-B alloys exhibited faster crack growth rate.

Dubey *et al.* [Dubey (1999)] studied investigated the fatigue response of as-cast Ti-64-0.5B alloy by three-point bending test at RT. They reported that fatigue striations were much less visible on the fatigue fracture surface of the as-cast Ti-64-0.5B alloy compared with the Ti-64 alloy. The fatigue crack growth of the as-cast Ti-64-0.5B alloy was not affected noticeably by the elastic bridging or the toughening from the crack bridging of the TiB whiskers.

Schwendiman [Schwendiman (2007)] studied the fatigue properties of PM Ti-64-1B rolled plate material. His study concluded that the PM rolled Ti-64-1B alloy outperformed the PM Ti-64 ELI and Ti-64 forged plates during the HCF tests. Ca, Fe, Si and Mg rich inclusions were suggested to be the dominant failure initiation sites based on the fractography and energy dispersive spectroscopy (EDS) analysis of the specimens exhibiting the lowest fatigue lives. Overall, Schwendiman's study suggested that the RT fatigue resistance of the PM rolled Ti-64-1B alloy is at least as good as the base line Ti-64 alloy.

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C. Creep Background

Creep is defined as the time-dependent deformation of a material at constant stress and constant temperature [Dieter, 1986]. Constant-load creep testing is performed when a specimen is placed under constant load while maintaining a constant temperature, usually greater than $0.4 T_m$ (melting temperature of the material). Figure 2.13 illustrates the typical shape of a creep curve. During a creep test, strain is measured using an extensometer and plotted versus time. The slope of the curve is defined as the creep rate. The curve consists of three regions. Following an instantaneous elongation of the specimen, the creep rate decreases with time. This region on the curve is considered primary creep. Then the creep rate reaches essentially a steady state in which the creep rate changes little with time. This region is referred to as the secondary creep. The constant creep rate is due to a competing process between strain hardening and recovery [Garofalo (1965)], which can be used to suggest the secondary-creep dominant deformation mechanism. Finally, the creep rate increases rapidly until fracture occurs. In general, creep behavior depends on stress and temperature, where the steady-state creep rate increases with increasing stress and/or increasing temperature.

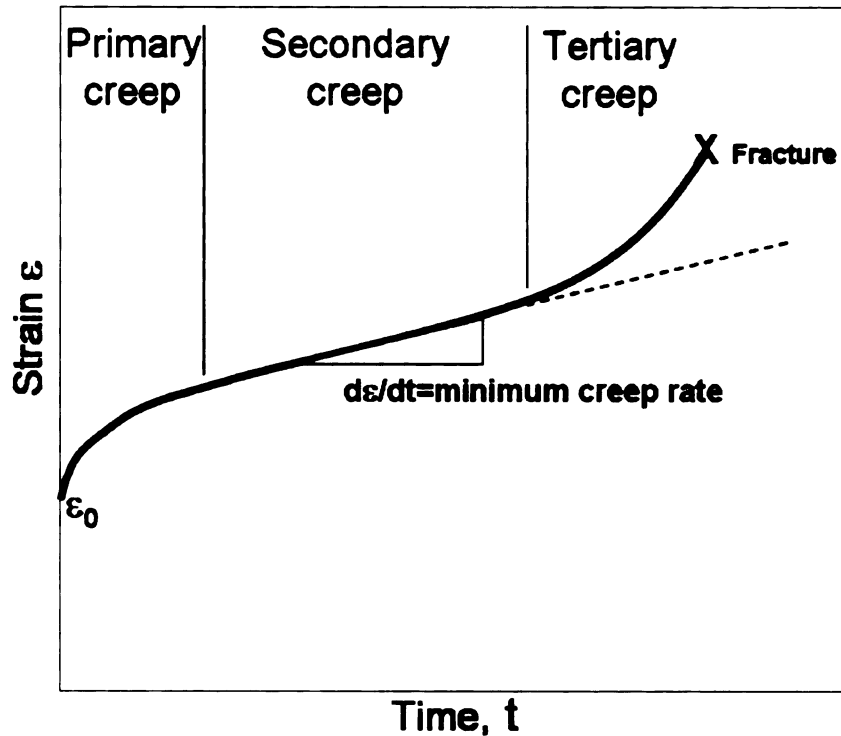


Figure 2.13. Idealized creep curve showing the three regimes of creep.

Extensive research on the creep behavior has led to the development of mechanisms for creep deformation. Major creep deformation mechanisms include diffusion creep, dislocation creep, and grain boundary sliding. Multiple creep mechanisms are often activated at the same time. If the creep mechanisms are operating independently, the fastest mechanism will dominate the creep behavior. If the creep mechanisms are operating in sequence, the slowest mechanism will control the creep deformation [Dieter (1986)].

1. Diffusion Creep

Diffusion creep occurs at $T/T_m > 0.4$ for $\sigma/G < 10^{-4}$, in which σ is the creep stress and G is the shear modulus of the material. This mechanism involves vacancies and

interstitials flow through a crystal under the influence of applied stress [Dieter (1986)]. This category includes Nabarro-Herring [Nabarro (1948), Herring (1950)] creep and Coble [Coble (1963)] creep.

In Nabarro-Herring creep, creep process is controlled by stress-directed atomic diffusion through the crystal lattice. Stress causes a change of chemical potential of the atoms on the grain surface such that vacancies flow from grain boundaries experiencing tensile stresses to those experiencing compressive stresses. Correspondingly, atoms flow in the opposite direction and leads to elongation of the grain. The steady state creep rate for Nabarro-Herring creep is given by:

$$\dot{\epsilon}_s = K_1 \frac{D_{SD}}{d^2} \frac{\Omega \sigma}{kT} \quad (2.1)$$

where K_1 is a constant, D_{SD} is the coefficient of lattice self diffusion, d is the average grain diameter, Ω is the volume of a vacancy, and k is Boltzmann's constant [Evans and Wilshire (1993)]. Nabarro-Herring creep is commonly observed at low stress and high temperature creep conditions [Evans and Wilshire (1993)].

In Coble creep, the driving force is the same as that for Nabarro-Herring creep, but the grain-boundary diffusion predominates. Coble creep is described by

$$\dot{\epsilon}_s = K_2 \frac{D_{GB}}{d^3} \frac{\delta \Omega \sigma}{kT} \quad (2.2)$$

where K_2 is a constant and δ is the grain boundary spacing. This mechanism is suggested to be dominant at low stress levels of the same magnitude of Nabarro-Herring creep, but at lower temperatures.

2. Dislocation Creep

Dislocation creep occurs for $10^{-4} < \sigma/G < 10^{-2}$, in which dislocations move and overcome barriers by thermally assisted mechanisms involving the diffusion of vacancies or interstitials [Dieter (1986)]. At elevated-temperature, if a gliding dislocation is held up by an obstacle, climb process may assist it to surmount the obstacle, allowing it to keep gliding until the next obstacle where the process is repeated [Weertman (1956)]. The glide process generates almost all of the strain but climb is the rate controlling in glide-climb processes since climb process is governed by atomic diffusion [Dieter (1986)]. In the dislocation creep regime, steady state creep is obtained when there is a balance between strain hardening and thermal recovery due to the rearrangement and annihilation of dislocations [Dieter (1986)]. At $T/T_m > 0.5$ and $10^{-4} < \sigma/G < 10^{-2}$, the steady state creep rate of a material is usually described using a power law relation:

$$\dot{\epsilon}_s = A \sigma^n \exp\left(\frac{-Q}{RT}\right) \quad (2.3)$$

where A is a constant, σ is the applied stress, n is the creep exponent, Q is the activation energy, R is the universal gas constant, and T is the absolute temperature.

This secondary creep mechanism was dominant for the applied temperature and stress range chosen in the current study of B-modified Ti alloys.

3. Grain Boundary Sliding

Grain boundary sliding involves the relative movement between grains in a polycrystalline metals and it is a shear process occurs in the direction of the grain boundary [Dieter (1986)]. In general, there is little strain exhibited within the grains.

Grain boundary sliding does not have a significant contribution to steady-state creep, but it is important in initiating intergranular fracture [Dieter (1986)]. However, Raj and Ashby [Raj (1971)] suggested that grain boundary sliding is required to maintain grain continuity during diffusional flow mechanisms.

Smaller grain size usually increases the amount of grain boundary sliding since the total number of grain boundaries in the material increases, therefore providing more chances for slip to occur between grains.

4. Creep Stress Exponents and Apparent Activation Energy

The values of creep stress exponents (n) and apparent activation energies (Q_{app}) can be used to suggest the dominant mechanisms during steady-state creep. The n and Q_{app} values are usually calculated using equation 2.3 after the minimum creep rate is determined from the creep strain versus time curve. The creep exponent is determined by applying a power law curve fit to the log-log plot of minimum creep rate versus stress, while the activation energy is determined from the slope of the least squares linear curve fit to the plot of the natural log of the minimum creep rate versus $1/T$.

In general, creep exponent values range from one to five for pure metals and disordered alloys [Evans and Wilshire (1985)]. Values close to unity suggest that vacancy diffusion is the dominant mechanism, indicating a mechanism of Nabarro-Herring or Coble creep. When Nabarro-Herring creep is dominant, Q_{app} values are found to be close to those for lattice self diffusion. When Coble creep is dominant, Q_{app} values are typically 0.5 – 0.6 of those for lattice self diffusion due to diffusion of vacancies through the grain boundaries.

For grain boundary sliding, n values are approximately two, and Q_{app} values are close to those of lattice self diffusion [Garafalo (1965), Hertzburg (1996)].

When dislocation creep is dominant, n values usually range from three to eight [Evans and Wilshire (1985)]. In this regime, activation energies correspond closely with those of lattice self diffusion.

Although creep exponents and apparent activation energies can be used to suggest the dominant creep mechanism, they are not always reliable especially when the investigated material is not a pure metal. Other techniques need to be used to verify the creep mechanism. TEM analysis on creep-deformed samples is typically used to observe dislocation activity in the microstructure. Grain boundary sliding may be demonstrated by making a fiducial mark on a polished and etched sample surface and observing the shear offset where the mark crosses grain boundaries.

5. Creep of Ti-B Alloys

Rhee *et al.* [Rhee (2003)] studied the creep behavior of a TiB reinforced Ti-6242S alloy produced by the blended elemental PM process. The creep conditions were from 550 – 675°C under a constant stress of 100 – 300 MPa in air. The TiB phase was found to significantly increase the creep resistance as it induced a strong retardation effect on the dislocation movement at low temperature and high stress. However, at high-temperature and lower-stress conditions, the retardation effect of the TiB reinforcement was reduced, essentially rendering the TiB whiskers irrelevant in the creep process. The creep stress exponent was determined to be 5.2 in this alloy, suggesting dislocation creep to be the dominant secondary creep mechanism.

Kawabata *et al.* [Kawabata (2004)] investigated the creep behavior of TiB-reinforced Ti in the β -phase field (1200°C) and observed that the reinforced Ti demonstrated greater creep resistance than the monolithic Ti for all the studied stress levels. They also suggested that if the misfit strain between the TiB reinforcement and the Ti matrix is accommodated by diffusion immediately, the load transfer effect is eliminated.

The elevated-temperature compressive flow properties of a TiB-reinforced Ti alloy as a function of temperature, strain rate, and the TiB whisker volume fraction was studied by Kumari *et al.* [Kumari (2004)]. The flow stress was found to increase with increasing strain rate and decreasing temperature. A higher volume fraction of TiB whiskers also increased the flow stress at a given temperature and strain rate.

CHAPTER 3

EXPERIMENTAL PROCEDURE

This chapter describes the experimental and analytical methods employed to carry out this research. Descriptions are given for the material processing details, sample preparation, microstructural characterization, equipment and methods used for the tensile, creep, *in-situ* creep, fatigue, and *in-situ* fatigue testing.

A. Material Processing

1. As-cast Ti-64-xB and Ti-6242S-xB Alloys

Castings of 70 mm diameter and 500 mm length were produced for Ti-64-xB and Ti-6242S-xB alloys at Flowserve Corporation, Dayton, Ohio via induction skull melting (Figure 3.1). The induction coils are labeled in Figure 3.1a. The melt was then poured into a crucible and slow cooled to RT, see Figure 3.1b. All castings were hot isostatically pressed (HIPped) at 900°C and 100MPa for two hours to remove porosity during the solidification process (Figure 3.2). This condition represented the as-cast state.

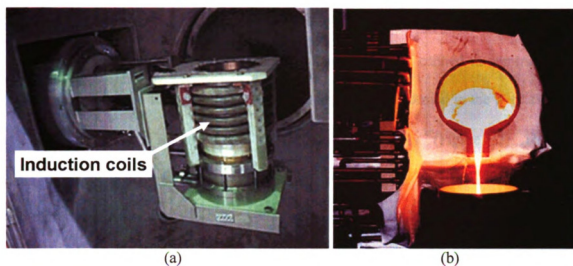


Figure 3.1. Induction Skull Melting Process. (a) The melting furnace, and (b) pouring of the melt into the crucible. (Courtesy: Scott Reed, FlowServe Corp.)

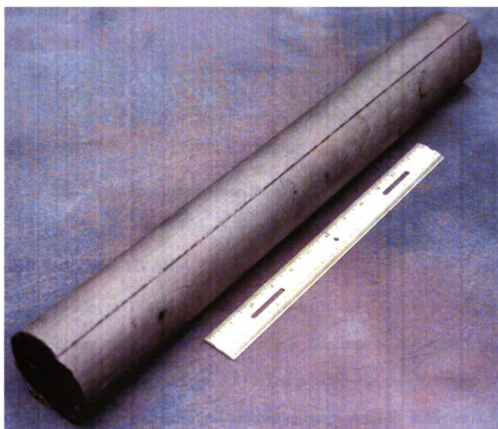


Figure 3.2. Cast ingot of Ti-64-xB or Ti-6242S-xB (70 mm diameter HIPped at 900°C/100 MPa for 2 hrs). The ruler length is 12 in (~30 cm).

2. Cast-and-extruded (CE) Ti-64-xB alloys

A portion of each Ti-64-xB casting was sectioned and canned in commercially-pure Ti in preparation for extruding. The extrusions were performed in the β -phase field at 1100°C using a 12:1 reduction ratio and the strain rate was 1 s^{-1} (Figure 3.3). The final diameter of the extruded rods was approximately 20mm and this represented the cast-and-extruded state.

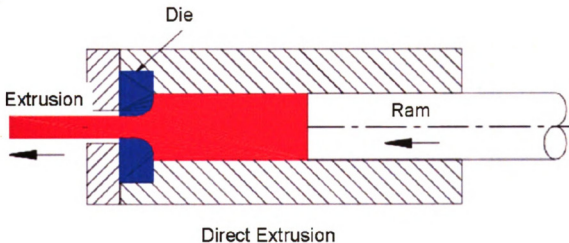


Figure 3.3. Diagram showing the extrusion process.

3. PM Rolled Ti-64-1B Alloy

The pre-alloyed Ti-64-1B (Ti-6Al-4V-1B-0.05C-0.11O) powder was made by induction skull melting followed by inert gas atomization in Crucible Research Corporation (Pittsburgh, PA) (Figure 3.4). The powder was then provided to FMW Composites Inc. for thermomechanical processing (steps outlined in Figure 3.5). The powder exhibited a martensite microstructure [Tamirisakandala (2005)]. The detailed atomization process was described by Dr. Youton [Yolton (2007)]:

"The Ti gas-atomization unit consists of a melting chamber, an atomization tower, and a cyclone collector. The boron is typically added as TiB_2 powder, and carbon is added as graphite powder. As the charge is melted, a skull forms at the crucible bottom and wall so the molten alloy is always contained in a solid skull of roughly the same composition. After the charge is fully melted and homogenized, it is bottom poured and atomized with high-pressure Ar gas. The atomization gas carries the powder to the cyclone where the powder is collected in a removable canister. After cooling under an Ar atmosphere to room temperature, the powder is removed for further processing."



Figure 3.4. Ti atomization tower and cyclone at Crucible Research Corporation used to make Ti-64-1B powder via pre-alloyed approach. The height of the room is about 5 m. [Yolton (2007)]

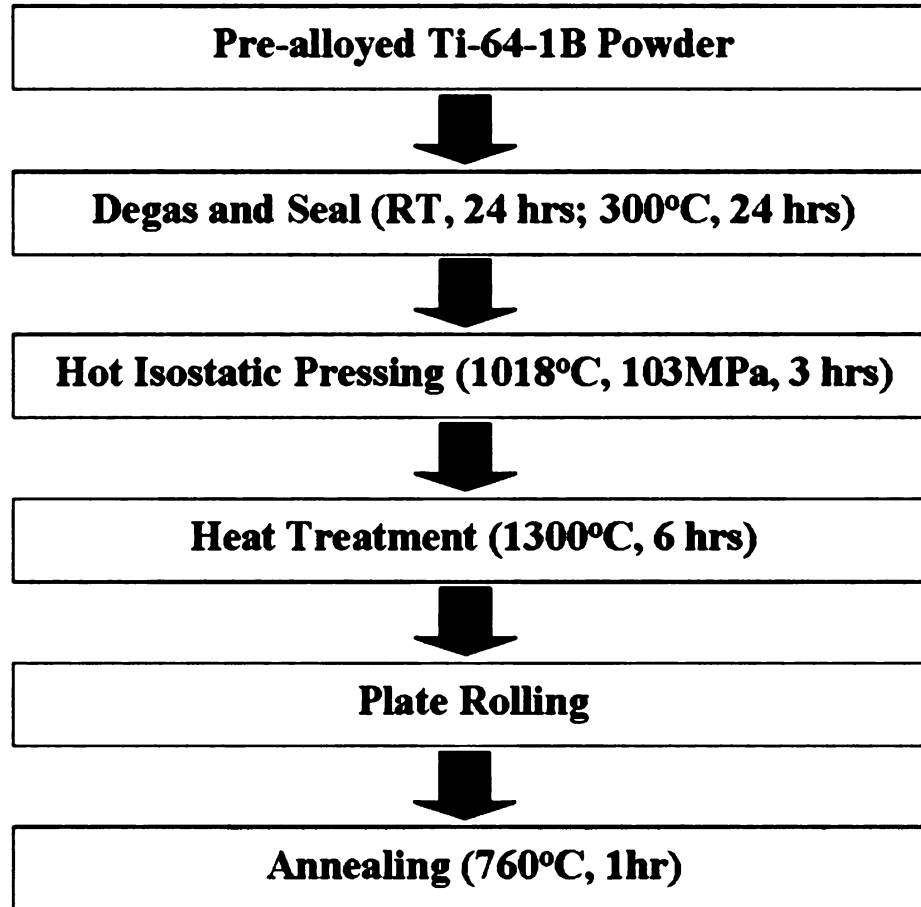


Figure 3.5. Thermomechanical processing steps for PM rolled Ti-64-1B alloy.

The Ti-64-1B powder was classified as -35 mesh ($<500\text{ }\mu\text{m}$ in diameter) [Yolton (2007)]. Degassing, sealing, HIPping, and heat treatment were used (Figure 3.5). Two Ti-64-1B powder compacts, weighing 1.6 kN (360 lbs) each were HIPped at 1018°C/103MPa for three hours followed by heat treatment at 1300°C for 6 hours at Bodycote in London, OH. The material was delivered to the Manufacturing Sciences Corporation (MSC) in Oak Ridge, TN for rolling. The MSC Oakridge facility used a 4-high reversing mill with 47 cm diameter rolls and 22.2×10^3 kN of separating force (Figure 3.6). The billet was loaded into a furnace and heated to 1100°C. Each billet was held for several hours in the furnace to ensure thermal soak, and was then transferred to

the rolling mill with a total transfer time of about 30 seconds (Figure 3.7). After the first round of 8 passes was conducted on the bill, the surface temperature dropped to 950°C (β transus temperature is $\sim 1000^\circ\text{C}$). The plate was then reheated to 1100°C. The heating-rolling process was done 3 times in total. The plate was cut into two pieces and flattened and stress relief annealed at 760°C for 1 hour and air cooled (Figure 3.8). This represented the PM rolled condition.



Figure 3.6. Rolling mill at Manufacturing Sciences Corporation (Oak Ridge, TN) facility. (Courtesy of Dr. Tamirisakandala)

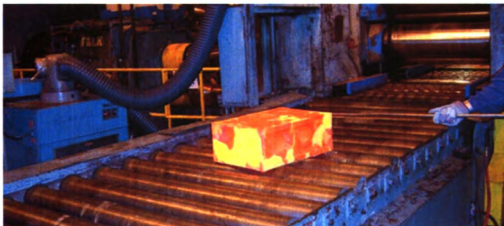


Figure 3.7. Ti-64-1B billet transferred from furnace to rolling mill. (Courtesy of Dr. Tamirisakandala)

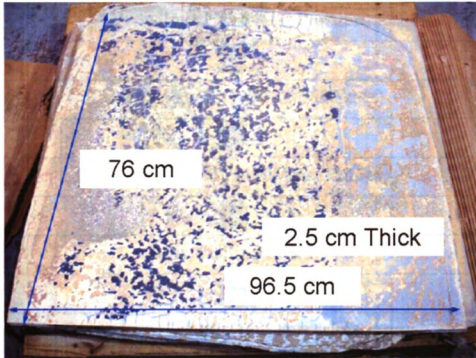


Figure 3.8. Final plates of PM rolled Ti-64-1B alloy. (Courtesy of Dr. Tamirisakandala)

4. PM Extruded Ti-64-1B Alloy

The pre-alloyed Ti-64-1B powder was the same as used for PM rolled Ti-64-1B alloy. One kilogram of powder was packed inside a thick-walled Ti-64 can of 70 mm diameter and 130 mm length. The can was sealed and heated to 1200°C, soaked for 3600 seconds, and compacted in an extrusion chamber using a blind-die. The compact was held at a pressure of 1400 MPa for 180 seconds followed by air-cooling to RT. A length reduction of approximately 30% occurred during compaction, and full density was achieved. The compact was then extruded at 1100°C in the β -phase field through a conical die using a 16:1 extrusion ratio (steps outlined in Figure 3.9). This represented the PM extruded condition.

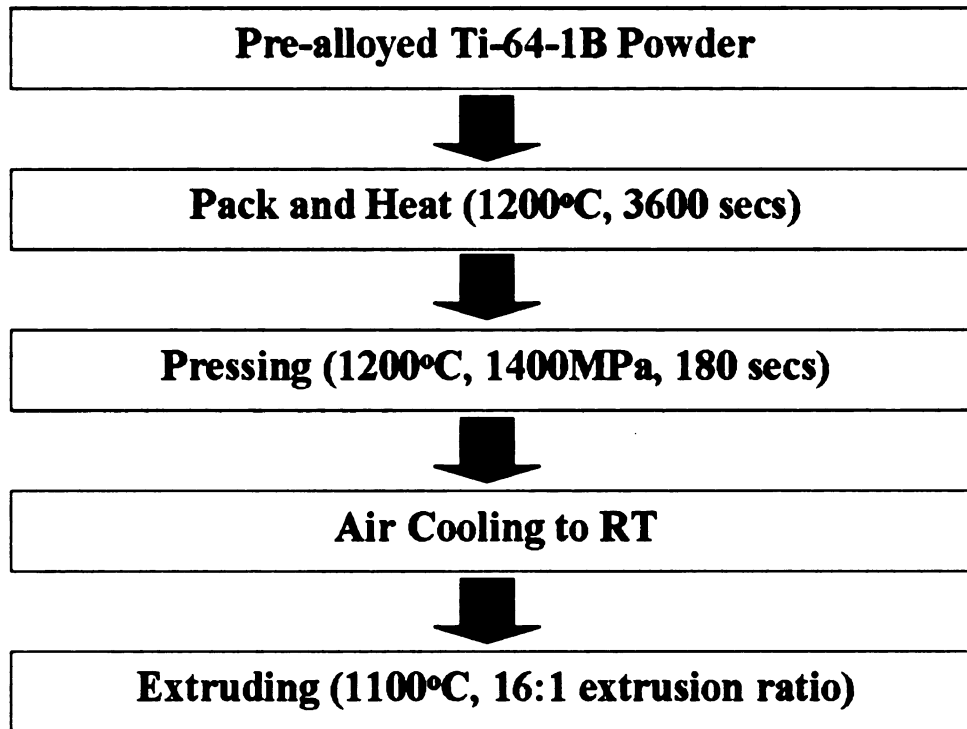


Figure 3.9. Thermomechanical processing steps for PM extruded Ti-64-1B alloy.

B. Microstructural Characterization

1. Chemical Composition Analysis

The bulk alloy compositions of the studied alloys were measured through Inductively Coupled Plasma Optical Emission Spectroscopy and Inert Gas Fluorescence. To determine the atomic percentages of Ti, Al, V, B, Mo, Zr, Sn, Mo, and Si in each phase, microprobe analysis was conducted on a JEOL JXA-8200 WD/ED combined microanalyzer operated at 10 kV and an emission current of approximately 55 nA. The low accelerating voltage used was chosen to minimize the overlap from the α + β phases and the TiB whiskers. The standards used were TiB₂ for Ti and B as well as pure Al and V metals for Al and V. The crystals used were LiFH for V and Ti, TAP for Al and a LDE₂ for B. The minimum level of detectable Si using the technique employed was estimated to be 0.01 wt.%, while for Mo and B the minimum detectable level was estimated to be 0.1-0.2 wt.%.

2. Metallography Preparation

The as-processed materials were prepared for imaging using SEM by sectioning the material into three different orientations as shown in Figure 3.10 (labeled face (F), transverse (T), and longitudinal (L)). The sections were made using a Buehler IsometTM low speed saw with a diamond blade. Sectioned samples were cleaned and mounted in KonductometTM using a Buehler Pneumat[®]-I mounting press forming 2.54 cm diameter mounts. The mounts were first collectively ground using SiC paper of successively finer grit size of 60 grit (1 minute), 240 grit (2 minutes), 400 grit (2 minutes), and then 600 grit

(2 minutes). They were then polished with 6 μm and 1 μm diamond paste suspensions for 10 minutes, respectively. Colloidal silica containing an average particle size of 0.05 μm was used for the final polishing (20 minutes) to produce a mirror finish. After polishing, the surface of the mounted samples was rinsed in acetone and ethanol to remove any residual colloidal silica solution.

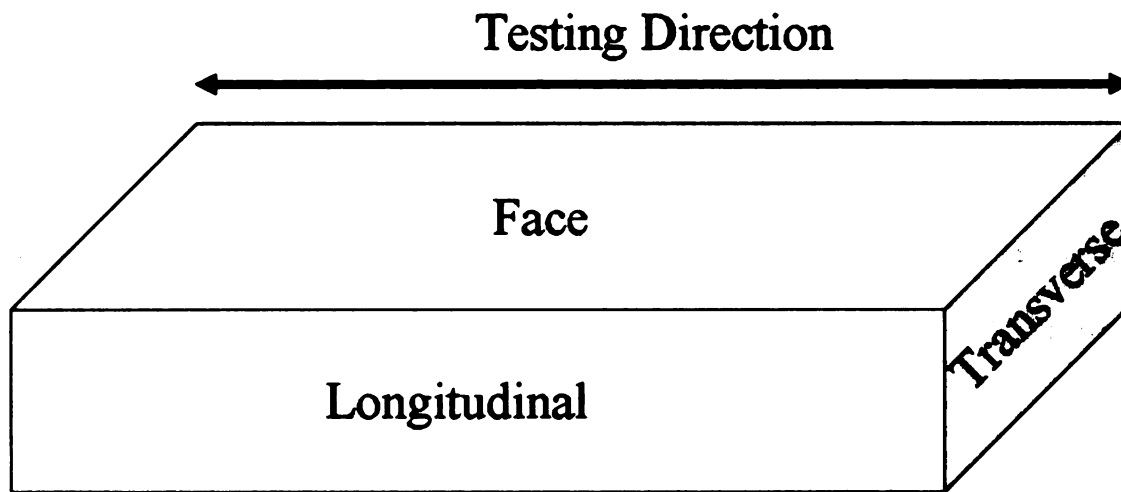


Figure 3.10. A schematic of the three different sheet orientations which were evaluated for the materials in the microstructural characterization.

3. Microscopy

3.1 Scanning Electron Microscopy

The alloy microstructure was characterized using Back Scattered Electron (BSE) mode on a CamScan 44FE field emission SEM. When imaging in the BSE mode, the contrast in the micrographs depends on the atomic number of the material element being bombarded with electrons in the SEM. The number of backscattered electrons escaping from the sample is proportional to the atomic number of the material being imaged

[Flegler (1993)]. Therefore, phases containing lighter elements exhibit darker contrast compared to phases containing heavier elements.

The BSE photographs were used to determine the grain size, colony size, lath width, and phase volume percentage. Phase volume percentage was determined using ImageJ image analysis software. This software can shade the pixels within the phases to different contrasts and then evaluate them with an area-fraction analysis macro. The macro counts the shaded pixels and divides that value by the total number of pixels in the image. The average equiaxed grain size and α -, β - lath widths of the studied alloys were determined using the Mean Line Intercept Method [Hilliard (1964)]. The lath widths of each alloy were measured perpendicular to the longitudinal axis of the laths for over 300 laths for each of the α and β phases. The average diameters (over 300 grains were measured) were multiplied by 1.74 to obtain the average grain size of equiaxed α grain.

3.2 Transmission Electron Microscopy

Transmission electron microscopy (TEM) analysis was performed using a Hitachi HF-3300 TEM/STEM at 300 kV. The samples were ground to a thickness of approximately 150 μ m then punched into 3mm diameter discs. The discs were then hand polished to approximately 100 μ m thick using 2400 grit silicon carbide paper and electropolished to perforation at a temperature of -15°C using a Struers (Westlake, OH) TenuPol-5 electropolishing unit at 28 volts. The electropolishing solution contained 60% methanol, 33.5% 2-Butoxyethanol, 6% Perchloric Acid, and 0.5% Glycerin.

4. EBSD Analysis

Electron backscatter diffraction (EBSD), performed using hardware and software manufactured by EDAX-TSL, Inc (Mahwah, NJ), was used to evaluate the microstructures. Spatially-resolved EBSD maps were acquired at 20-30 keV using a step size ranging between 0.2-1.0 μm on samples polished through colloidal silica (0.06 μm average particle diameter).

5. X-ray Diffraction Analysis

X-ray Diffraction (XRD) 2θ scans were collected from 20-120° at a step size of 0.0167° using a X'Pert Pro MPD diffractometer (PANalytical, Inc., Almelo, Netherlands) with copper K_{α} radiation. XRD pole figures on a 5° by 5° grid were acquired using a PTS goniometer (Scintag, Inc. Sunnyvale, CA) with chromium K_{α} radiation over a tilt range from 0-75°.

C. Mechanical Testing

1. Sample Configuration

1.1 Tensile, Creep and Fatigue Experiments

The alloy castings, plates, and extrusion bars were cut into flat dog bone specimens whose dimensions measured 125 mm in length with a 12.5 mm wide reduced gage section. For the castings and extrusion bars, samples were cut along the axis of the castings or extrusion bars (Figure 3.11a). For the plates, samples were cut along the rolling direction (Figure 3.11b). Figure 3.12 shows the real dimension of the dog bone samples.

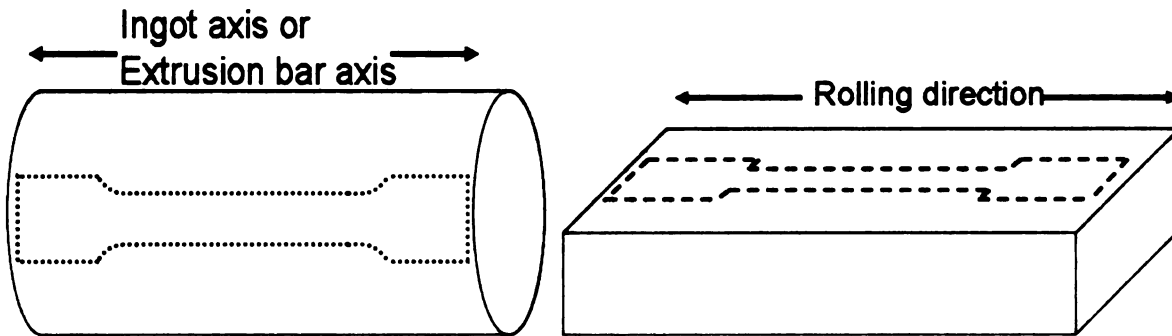


Figure 3.11. Sample cutting direction of the (a) castings and extrusion bars, and (b) rolled plates.

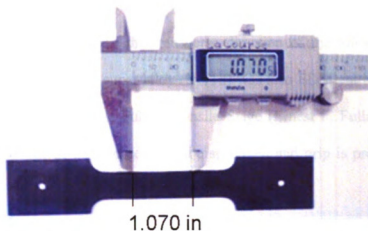


Figure 3.12. Photo of the sample used for tensile, creep, and fatigue testing.

1.2 *In-situ* Experiments

For the *in-situ* tensile, creep, and fatigue experiments, specially-designed specimens containing a gage section of 10 mm in length were used. Prior to testing, *in-situ* specimens were glued to a metallic platen and polished through a 0.06 μm colloidal silica finish using an automatic polishing machine, which allowed for optimal analysis using BSE SEM. An image of the specimen geometry used is provided in Figure 3.13.

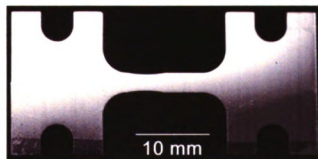


Figure 3.13. A photograph of the dog bones used for *in-situ* tensile, creep, and fatigue testing.

1.3 Tensile Experiments in Transverse Direction

In order to evaluate the anisotropy of the studied alloys, samples of special dimension were EDM cut along the transverse direction of the alloy bars or plates. Special grip made of Ni-based super alloy (IN718) was also EDM cut to fit the samples for tensile tests using the Ernest Fullam Tensile Stage (Ernest F. Fullam, Inc., Clifton Park, NY). An image of the geometry of the specimen and grip is provided in Figure 3.14.

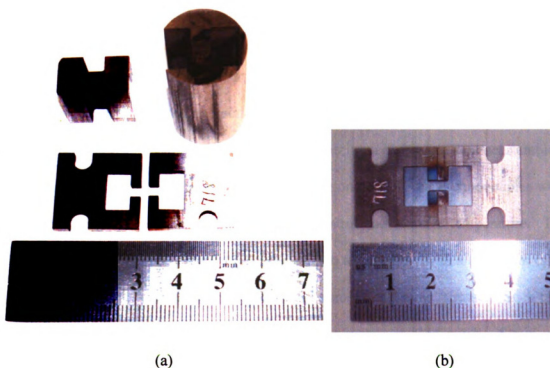


Figure 3.14. (a) A sample EDM cut from the Ti-64-1B alloy extrusion bar and the IN718 grip, and (b) Sliced sample fit into the IN718 grip.

2. Conventional Mechanical Testing

2.1 Tensile Testing

Room temperature (RT) and Elevated temperature (455°C) tensile experiments were performed in laboratory air, using a horizontal servo-hydraulic test machine (Figure 3.15 and Figure 3.16). The specimens were precisely aligned, ensuring truly uniaxial loading, using uniquely-designed friction grips. The displacement was controlled by an MTS Flex Test SE Controller using Basic Testware software. Strain was monitored using an alumina-rod high-temperature extensometer, with a 12 mm gage length, attached to the gage section of the specimen. The heating unit contained eight quartz lamps, two banks of four lamps each, oriented perpendicular to the loading axis, one above and one below the specimen. A 25 mm section in the center of the specimen was uniformly heated ($\pm 5^\circ\text{C}$ of the target temperature), while the specimen ends were kept near ambient temperature through the use of the water-cooled friction grips. Four thermocouples were attached to the gauge section of the samples to control and monitor temperature. The 455°C tensile tests incorporated a 900 second ramp to temperature with a minimum of 20 minutes soaking time before loading in order to minimize the thermal stresses. Such tests were conducted using a displacement rate of approximately 0.0254 mm/s. Strain was monitored using an alumina-rod high-temperature extensometer, with a 12mm gage length, attached to the gage section of the specimen. The YS, UTS, ϵ_f were determined through the tensile data. The Young's modulus was calculated using linear regression methods on the elastic portion of the stress/strain data. The average tensile properties (at least 2 tests per condition) were reported at RT and 455°C.

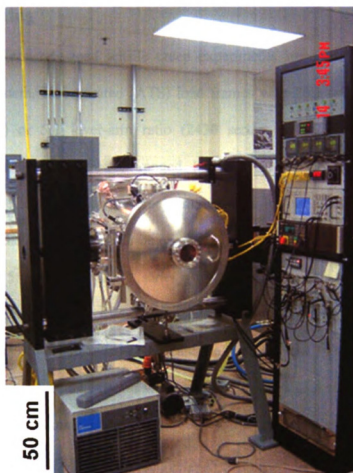


Figure 3.15. Servo-hydraulic testing frame used for tensile and fatigue testing.

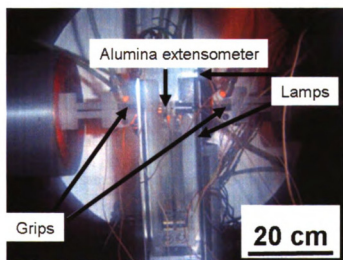


Figure 3.16. Horizontal servo-hydraulic testing frame showing a gripped sample with an attached extensometer and two banks of quartz lamps.

2.2 Creep Testing

Open-air, constant load tensile creep experiments were performed on vertical Applied Test System, Incorporated (ATS) load frames with 20:1 lever-arm ratio (2410 and 2710 series) or 5:1 lever-arm ratio (2430 series), see Figure 3.17. The test temperatures and applied stresses ranged between 400-565°C and 138-600 MPa, respectively. The load cells of the creep frames were located above the weights (see Figure 3.17), so the specimens could be examined at loads up to 20 times (for 2410 and 2710 series) or 5 times (for 2430 series) the maximum capacity of the load cell. Although the experiments were constant load, in most cases the reduction in cross-sectional area was not sufficient to significantly alter the stress. Therefore, the stresses were assumed to be constant. Strain was measured using a 25.4 mm gage length ATS high-temperature extensometer attached directly to the specimen's gage length. Linear variable differential transformers, with a strain resolution of 0.0001, were used to track the displacement of the extensometers and output the data to the computerized data acquisition system (Labview Version 7.1). The data acquisition system collected load, strain, and time data at desired time intervals.

Specimen temperatures were monitored by three chromel-alumel type K thermocouples located within the reduced section of the specimen. Two thermocouples were spot-welded directly to the gage section of the sample. Another thermocouple was placed near the gage of the sample. Targeted temperatures were maintained within $\pm 3^\circ\text{C}$. The experiments were conducted such that the specimens were soaked at the creep temperature for at least 60 minutes prior to applying load in order to minimize the thermal stresses. After the creep strain had proceeded well into the secondary regime and

a steady-state creep rate was achieved, either the load or temperature was changed or the creep test was discontinued. The tested specimens were cooled under load to minimize recovery of the deformed structures. Selected specimens were taken to failure. Calculated steady-state creep rates were determined for each stress/temperature combination. These values were then used to determine creep stress exponents and apparent activation energies.

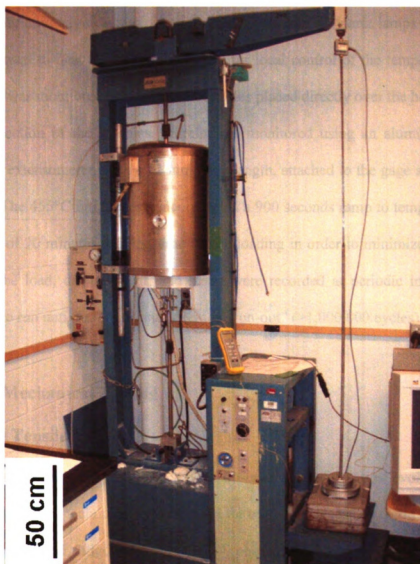


Figure 3.17. An ATS creep frame used to test the alloys in this work.

2.3 Fatigue Testing

Iso-thermal fatigue tests at 455°C were conducted on the same test frame as that described in Section 2 (Tensile testing) of this chapter. All tests were conducted in load control with a stress ratio (R) of 0.1. The test frequency was 5 Hz. The maximum applied stress ranged from 250-700 MPa, and the temperature was 455°C. The load and temperature were controlled by an MTS Flex Test SE Controller using Multipurpose Testware software. The specimens were precisely aligned, ensuring truly uniaxial loading, using uniquely-designed friction grips. Two sets of quartz lamps (eight lamps total) were used to heat the sample, allowing for local control of the temperature. The temperature was monitored by four thermocouples placed directly over the heated zone in the gauge section of the samples. Strain was monitored using an alumina-rod high-temperature extensometer, with a 12 mm gage length, attached to the gage section of the specimen. The 455°C fatigue tests incorporated a 900 seconds ramp to temperature with a minimum of 20 minutes soaking time before loading in order to minimize the thermal stresses. The load, displacement, and strain were recorded at periodic intervals. All samples were run until failure or until deemed “run-out” (>1,000,000 cycles).

3. *In-situ* Mechanical Testing

3.1 *In-situ* Tensile Testing

In-situ technique is the best way to observe the deformation evolution on the sample surface. This technique was also used for tracking the surface deformation during the tensile, creep and fatigue experiments. A photo of the testing stage used (built by Ernest F. Fullam, Incorporated, Clifton Park, NY) is given in Figure 3.18. The *in-situ*

tensile tests were performed at temperatures between 400-565°C inside a CamScan 44FE SEM or a XL-30 (FEI, Inc.) field emission gun SEM. Temperature was controlled using a constant-voltage power supply which provided output to a 6 mm diameter tungsten-based heater located just below the gage section of the sample. An open-bath, closed-loop chiller was used to circulate distilled water at RT through copper tubes to prevent the tensile stage from overheating. Two thermocouples were spot-welded to the gage section of each sample. After the sample's gage-section temperature reached the desired value, 30 minutes soaking time was given to stabilize the thermal stress prior to applying load. The displacement rate for the in-situ tensile tests was also 0.0254 mm/s. Before each test, the geometric data were put into MTESTW data acquisition and control software (Admet, Inc., Norwood, MA), so a stress-displacement curve was showing up during the tensile tests. It is noted that the displacement data acquired during the experiments comprised that of the sample as well as the gripping fixtures. SEM images were taken before loading and at certain stress levels after stopping the tensile tests to record the surface deformation process.

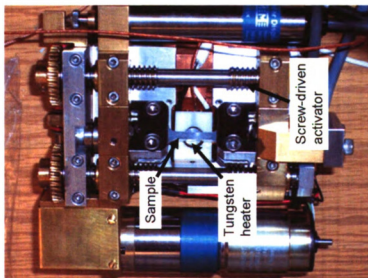


Figure 3.18. The Ernest Fullam *in-situ* tensile stage.

3.2 *In-situ* Creep Testing

In-situ tensile-creep experiments were conducted on the same stage as described in 2.1. Flat dogbone-shaped samples, with gage dimensions of 3.0 mm wide by 2.5 mm thick by 10 mm long, were prepared. After the temperature of the samples reached the desired creep temperature, a period of 30 minutes was given to stabilize the thermal stress prior to applying load. The load, which was measured using a load cell with a capacity of 4.448 kN, was applied at 3.7 N/s until reaching the desired creep stress. The tests were considered constant load where the stress fluctuation varied ± 3.0 MPa. The displacement data acquired during the experiments comprised that of the sample as well as the gripping fixtures. Thus the displacement values reported do not represent the sole displacement of the reduced gage section of the sample. BSE SEM images were taken before loading and at periodic displacements throughout the creep experiments without interrupting the experiment. The pressure in the SEM chamber never exceeded 10^{-4} Pa, and therefore oxidation did not detrimentally affect the SEM imaging.

3.3 *In-situ* Fatigue Crack Growth Testing

In-situ fatigue crack growth experiments were also performed using the same stage. In these tests, a 0.5 mm notch was EDM cut into one side of the gage length. Two sets of thermocouples were spot welded to the specimen's gage section to monitor the temperatures. The tests were performed with a total cycle time of approximately 40 minutes and a stress ratio of 0.1. The pressure in the SEM chamber was about 10^{-6} torr and secondary electron (SE) images were acquired before, during, and after the experiments. The experiments were never stopped or paused, thus the images acquired

during the experiments were taken either during the loading or unloading portions of the cycles.

3.4 Tensile Testing in Transverse Direction

Due to the geometry limitation of the extruded alloy bars and rolled alloy plates, tensile properties perpendicular to the extrusion direction or rolling direction have not been evaluated before. By designing special sample and grip (as described in 1.3), the *in-situ* stage was used for evaluating the anisotropy of the extruded alloy bars and rolled alloy plates, see Figure 3.19.

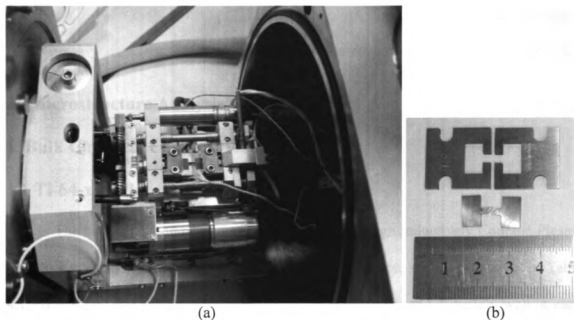


Figure 3.19. (a) A sample on tensile stage ready to put into the XL-30 SEM chamber, and (b) the grip and broken sample after a tensile test.

CHAPTER 4

RESULTS

This chapter presents the results obtained from the characterization of the microstructure, the mechanical properties, and the deformation process. It is separated into subsections of microstructure analysis, tensile behavior, fatigue behavior, fatigue crack growth behavior, and creep behavior. Each subsection contains two different alloy systems, namely the Ti-64-xB alloy system and the Ti-6242S-xB alloy system. The aim of this chapter is to identify the processing-microstructure-properties relationship for the Ti-64-xB and Ti-6242S-xB system. The analysis of the results will be discussed in Chapter 5.

A. Microstructure Analysis

1. Bulk Chemical Compositions

1.1 Ti-64-xB Alloy System

The bulk alloy compositions were measured through Inductively Coupled Plasma Optical Emission Spectroscopy and Inert Gas Fluorescence, which are shown in Table 4.1. It is noted that the targeted compositions were maintained adequately well in each alloy. No post-processing heat treatments were performed prior to the tensile, creep, and fatigue testing.

Table 4.1. Bulk Compositions of the Ti-64-xB Alloys

Alloy	Processing	Al, wt.%	V, wt.%	B, wt.%	Fe, ppm	O, ppm	N, ppm	C, ppm	H, ppm
Ti-64	as-cast	6.6	4.1	0	2300	1800	100	200	80
Ti-64-0.1B	as-cast	6.0	4.0	0.1	1300	1500	100	200	50
Ti-64-1B	as-cast	5.9	4.0	1.0	1300	1800	200	200	90
Ti-64	cast-and-extruded	6.1	4.1	0	2000	1770	90	N/A	N/A
Ti-64-0.1B	cast-and-extruded	6.0	4.0	0.1	1400	1380	80	N/A	N/A
Ti-64-1B	cast-and-extruded	5.9	3.9	0.8	1400	1600	70	238	N/A
Ti-64-1B	PM rolled	6.4	4.1	1.0	500	1150	120	560	68
Ti-64-1B	PM extruded	6.5	3.6	1.1	360	1250	100	1000	100

*Ti was the balance in the composition.

1.2 Ti-6242S-xB Alloy System

The bulk alloy compositions of the Ti-6242S-xB alloy system were measured through Inductively Coupled Plasma Optical Emission Spectroscopy and Inert Gas Fluorescence, which are shown in Table 4.2. Targeted compositions were maintained quite well in each alloy. No post-processing heat treatments were performed prior to the tensile, creep and fatigue testing.

Table 4.2. Bulk Compositions of the As-Cast Ti-6242S-xB Alloys

Alloy	Al, wt.%	Sn, wt.%	Zr, wt.%	Mo, wt.%	Si, wt.%	B, wt.%	Fe, ppm	O, ppm	N, ppm	C, ppm	H, ppm
Ti-6242S	5.8	2.0	4.1	2.0	0.08	0	700	1500	40	100	10
Ti-6242S-0.1B	6.0	2.0	4.0	2.1	0.09	0.09	400	1400	100	100	70
Ti-6242S-0.4B	5.9	1.9	4.0	2.0	0.10	0.44	500	1600	100	200	40
Ti-6242S-1B	6.1	1.9	4.1	2.0	0.11	0.92	500	1800	100	100	80

*Ti was the balance in the composition.

2. Microstructures

2.1 Ti-64-xB Alloy System

2.1.1 As-cast and Cast-and-extruded Alloys

Backscattered electron SEM images of the Ti-64-xB alloys' microstructures are shown in Figure 4.1 for Ti-64-xB alloys. In general, there are three phases in the Ti-64-xB alloys: The TiB phase is the darkest, the α phase is gray, and the β phase is the brightest. The crystal structures of the α -Ti, β -Ti, and TiB phases are hcp, bcc and orthorhombic, respectively.

Figure 4.1. BSE SEM images of the alloy microstructures: (a) Ti-64 as-cast, (b) Ti-64-0.1B as-cast, (c) Ti-64-1B as-cast, (d) Ti-64 cast-and-extruded, (e) Ti-64-0.1B cast-and-extruded, and (f) Ti-64-1B cast-and-extruded alloys. In cast-and-extruded alloys, the extrusion direction was horizontal. The TiB phase is the darkest, the α phase is gray, and the β phase is the brightest.

Figure 4.1 (continued)

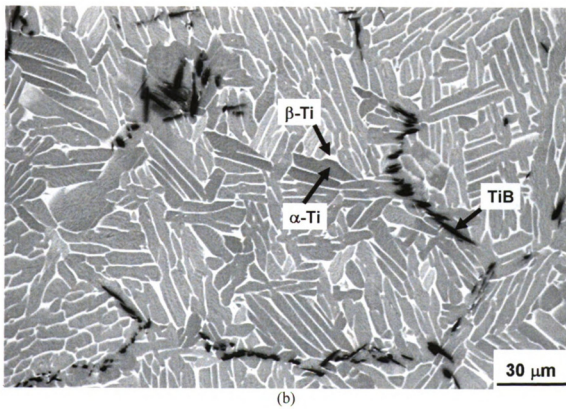
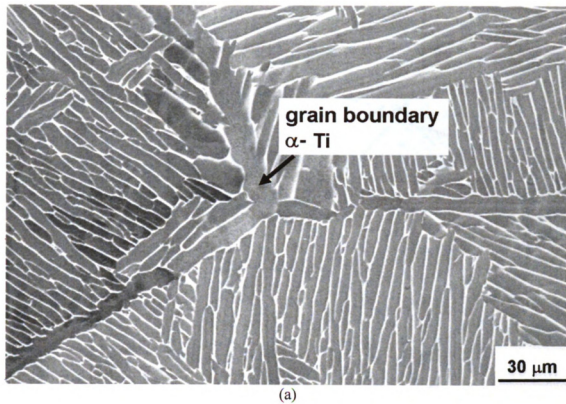
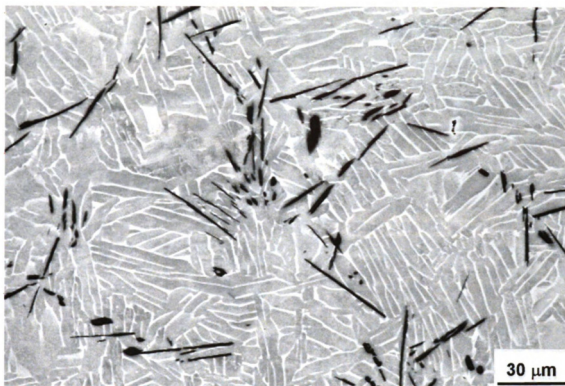
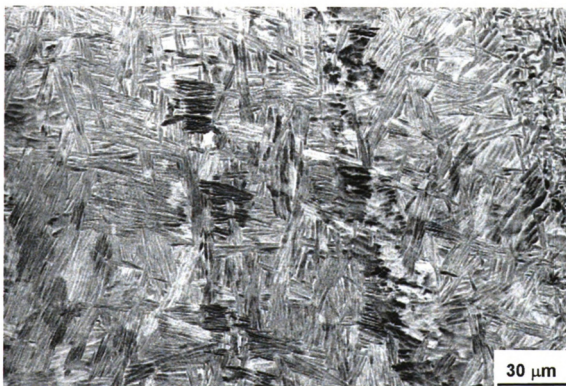


Figure 4.1 (continued)

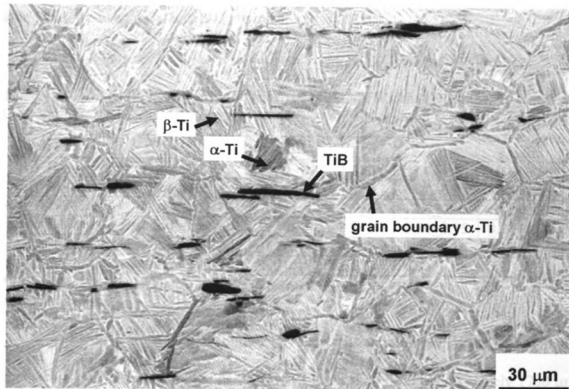


(c)

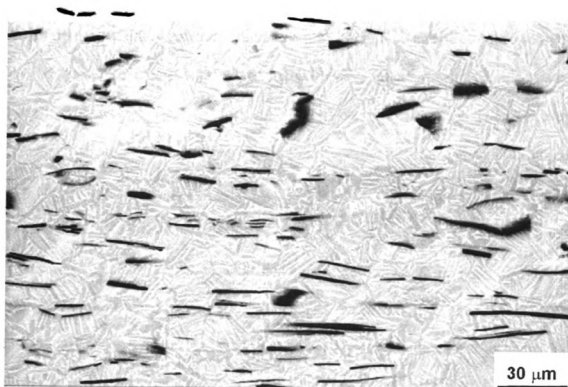


(d)

Figure 4.1 (continued)



(e)



(f)

Table 4.3 lists the average volume percents of the TiB and β phases for each alloy along with the average α - and β -lath widths.

Table 4.3. TiB and β Phase Vol.% (V_p) and α and β Lath Widths (λ) for the As-cast and Cast-and-extruded Ti-64-xB Alloys

Alloy	Processing	V_p TiB*	V_p β *	λ_α , μm	λ_β , μm
Ti-64	as-cast	0	12.8 $\pm$ 2.6	3.8 $\pm$ 1.1	0.6 $\pm$ 0.1
Ti-64-0.1B	as-cast	0.6 $\pm$ 0.3	14.4 $\pm$ 2.3	4.2 $\pm$ 0.5	0.8 $\pm$ 0.1
Ti-64-1B	as-cast	5.8 $\pm$ 1.8	15.5 $\pm$ 2.0	3.6 $\pm$ 0.8	0.7 $\pm$ 0.2
Ti-64	cast-and-extruded	0	21.2 $\pm$ 2.1	0.84 $\pm$ 0.15	0.19 $\pm$ 0.03
Ti-64-0.1B	cast-and-extruded	0.6 $\pm$ 0.1	19.2 $\pm$ 2.7	0.79 $\pm$ 0.14	0.13 $\pm$ 0.01
Ti-64-1B	cast-and-extruded	6.0 $\pm$ 1.4	18.5 $\pm$ 2.3	1.04 $\pm$ 0.18	0.20 $\pm$ 0.05

*The α phase was the balance in the microstructure.

As the BSE SEM images (Figure 4.1) show, the addition of 0.1B to Ti-64 refined the as-cast grain size (referred to as the prior- β grain size) from approximately 1700 μm to 200 μm and B addition eliminated the continuous grain boundary α network. The TiB phase formed a necklace structure around the prior- β grain boundaries in the as-cast Ti-64-0.1B alloy, see Figure 4.1b, which helped restrict grain growth [Tamirisakandala (2005)]. The average α -colony sizes of as-cast Ti-64, Ti-64-0.1B, and Ti-64-1B alloys were approximately 200 μm , 60 μm and 30 μm , respectively. The average prior- β grain sizes and α -colony sizes ranged between 30-70 μm and 10-20 μm , respectively, for the cast-and-extruded alloys where decreased grain sizes and α -colony sizes correlated with increasing B contents. Thus the extrusion procedure decreased the grain size and α -colony size more than adding 0.1 wt.% or 1 wt.% of B to the casting. The photomicrographs in Figure 4.1 show that higher B concentrations lead to higher TiB phase (darkest phase) volume percents. The volume percents of TiB in the Ti-64-0.1B

and Ti-64-1B alloys were approximately 0.6% and 6.0%, respectively. The volume percents of the β phase remained relatively constant ($13\% < V_p \beta < 16\%$) for each as-cast alloy. The volume percents of the β phase in the Ti-64-xB cast-and-extruded alloys ranged between 18-21%. The average α -lath width varied slightly with B concentration and was always between 3.6-4.2 μm for the as-cast alloys. The extrusion procedure significantly reduced the lath widths. For the cast-and-extruded alloys, the lath widths were approximately four times smaller than those for the as-cast alloys. Similar to the as-cast alloys, no significant effect of B was observed with respect to the lath widths for the cast-and-extruded Ti-64-xB alloys (see Table 4.3). For the Ti-64-1B alloys, the TiB whiskers ranged in length up to 50 μm and diameter up to 3 μm . As can be seen from Figures 4.1e and f, the TiB phase was aligned along the extrusion axis. This was evident in the TEM observations as well, see Figure 4.2.

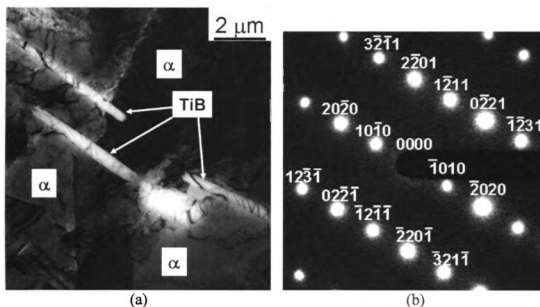
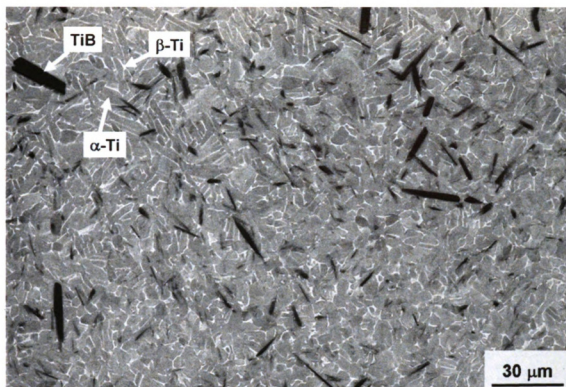


Figure 4.2. TEM data for the Ti-64-1B cast-and-extruded alloy (a) bright field image indicating the TiB phase was aligned in the extrusion direction and a (b) $[12\bar{1}6]$ SADP taken from the α -phase. (Image acquisition was assisted by Dr. Jane Howe of ORNL)

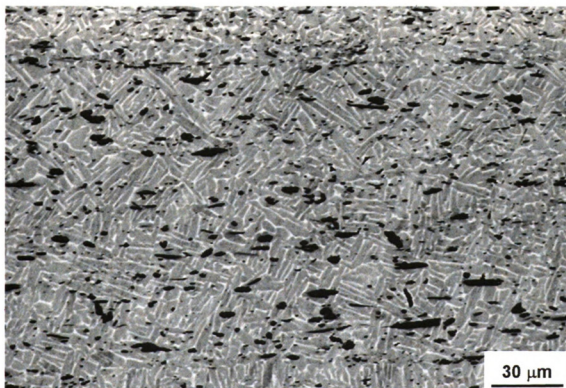
2.1.2 PM Processed Ti-64-1B Alloys

BSE SEM images of the alloy microstructure are shown in Figure 4.3 for PM processed Ti-64-1B alloys. Similar to the as-cast and cast-and-extruded Ti-64-1B alloys, there are also three phases in PM processed Ti-64-1B alloys: The TiB phase, the α phase, and the β phase. The TiB phase in the PM rolled alloy was randomly oriented. The TiB phase in the PM extruded alloy was aligned in the extrusion direction. However, the α -phase morphology was quite different from that in the as-cast or cast-and-extruded Ti-64-1B alloys. The PM rolled Ti-64-1B alloy exhibited a duplex microstructure, which contained both lenticular and equiaxed α phase. The PM extruded Ti-64-1B alloy exhibited a pure equiaxed microstructure and the equiaxed α grain size was much finer than that of the PM rolled Ti-64-1B alloy. The α grain diameter in PM extruded alloy was 3.6 μm compared with 7.4 μm in PM rolled alloy. In addition, the length of TiB whiskers in the PM rolled Ti-64-1B alloy varied more than in the other Ti-64-1B alloys, which were as-cast, cast-and-extruded, and PM extruded. One possible reason is that the rolling process introduced severe mechanical deformation and broke the TiB whiskers.

Figure 4.3. BSE SEM images of the PM processed Ti-64-1B alloy microstructures. In (d), the TiB phase was aligned in the extrusion direction (horizontal). In (e), the TiB phase is pointing out of the paper. The TiB phase is the darkest, the α phase is gray, and the β phase is the brightest.

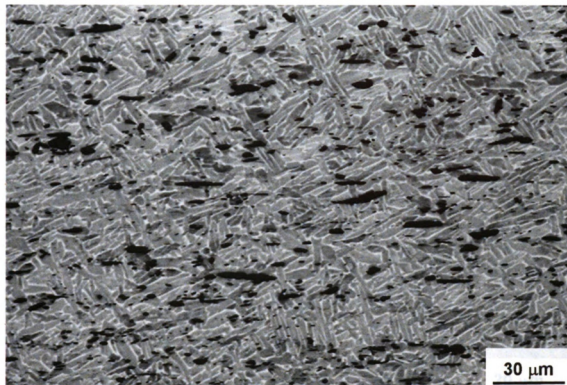


(a) PM rolled face

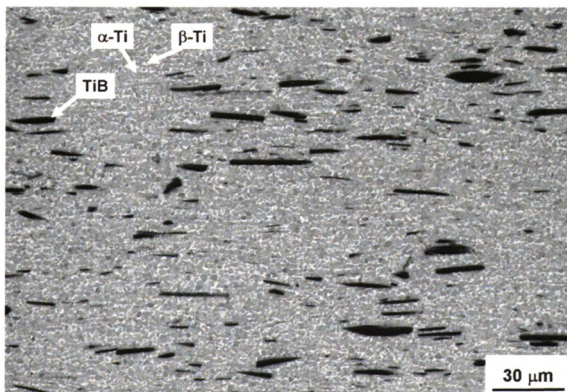


(b) PM rolled longitudinal

Figure 4.3 (continued)

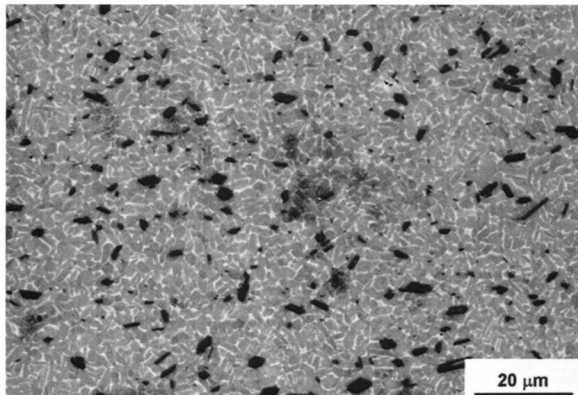


(c) PM rolled transverse



(d) PM extruded face

Figure 4.3 (continued)



(e) PM extruded transverse

Table 4.4 lists the average volume percents of the TiB and β phases for the PM processed alloy along with the equiaxed α grain size and average α - and β -lath widths. The volume percents of TiB in both the PM rolled and PM extruded Ti-64-1B alloys were approximately 6.0, which is the same as in the as-cast and cast-and-extruded Ti-64-1B alloys. The volume percents of the β phase is around 15%, which is close to the value for the as-cast Ti-64-1B alloy but about 3% lower than that for the cast-and-extruded Ti-64-1B alloy. The average α -lath width in the PM rolled Ti-64-1B alloy was about 2.7 μm , which was between that of the as-cast (3.6 μm) and cast-and-extruded (1.0 μm) Ti-64-1B alloys. The average grain size of equiaxed α phase was 3.6 μm in PM extruded Ti-64-1B alloy, which was significantly smaller than the α -grain size (7.4 μm) in PM rolled Ti-64-1B alloy.

Table 4.4. TiB and β Phase Vol.% (V_p), α Grain Size (d_α) and α , β Lath Widths (λ) for the PM Processed Ti-64-1B Alloys

Alloy	Processing	V_p TiB*	V_p β *	λ_α , μm	λ_β , μm	d_α , μm
Ti-64-1B	PM rolled	5.9 $\pm$ 1.4	15.5 $\pm$ 2.0	2.7 $\pm$ 0.5	0.4 $\pm$ 0.1	7.4 $\pm$ 0.7
Ti-64-1B	PM extruded	6.3 $\pm$ 1.3	14.9 $\pm$ 1.8	N/A	N/A	3.6 $\pm$ 0.8

*The α phase was the balance in the microstructure.

2.2 As-cast Ti-6242S-xB Alloy System

BSE SEM images of the alloy microstructure are shown in Figure 4.4 for as-cast Ti-6242S-xB alloys. The microstructure of this alloy system is similar to that for the as-cast Ti-64-xB alloy system as it contained the TiB, α , and β phases and the matrix contained the α + β lenticular microstructure.

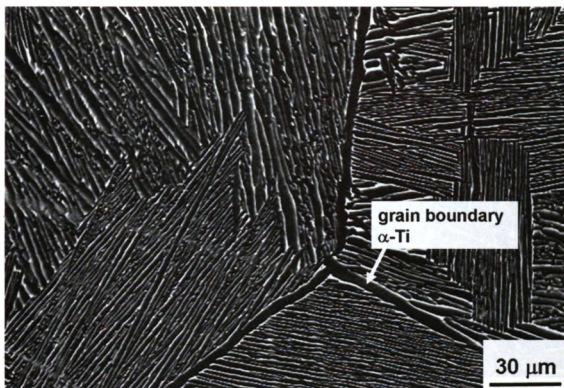
Table 4.5 lists the volume percents of the TiB, α , and β phases along with the average α -phase and β -phase lath widths in the as-cast Ti-6242S alloys.

Table 4.5. TiB and β Phase Vol.% (V_p) and α and β lath widths (λ) for the As-cast Ti-6242S-xB Alloys

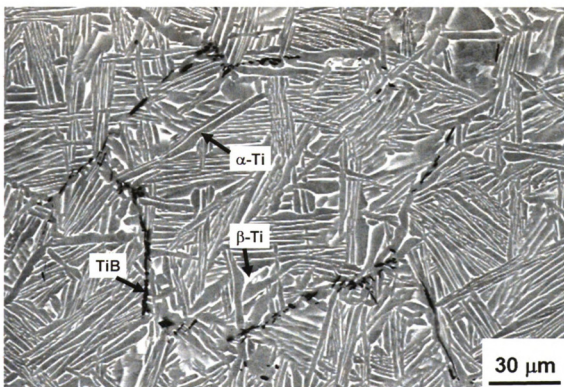
Alloy	V_p TiB*	V_p β *	λ_α , μm	λ_β , μm
Ti-6242S	0	22.5 $\pm$ 1.9	1.26 $\pm$ 0.15	0.35 $\pm$ 0.06
Ti-6242S-0.1B	0.74 $\pm$ 0.13	22.3 $\pm$ 0.9	1.43 $\pm$ 0.20	0.35 $\pm$ 0.06
Ti-6242S-0.4B	2.31 $\pm$ 0.12	22.1 $\pm$ 0.9	2.08 $\pm$ 0.43	0.38 $\pm$ 0.10
Ti-6242S-1B	5.42 $\pm$ 0.54	20.7 $\pm$ 0.8	2.21 $\pm$ 0.45	0.38 $\pm$ 0.09

*The α phase was the balance in the microstructure.

Figure 4.4. BSE SEM images of the as-cast Ti-6242S-xB alloy microstructure: (a) Ti-6242S, (b) Ti-6242S-0.1B, (c) Ti-6242S-0.4B and (d) Ti-6242S-1B alloys. The TiB phase is the darkest, the α phase is gray, and the β phase is the brightest.

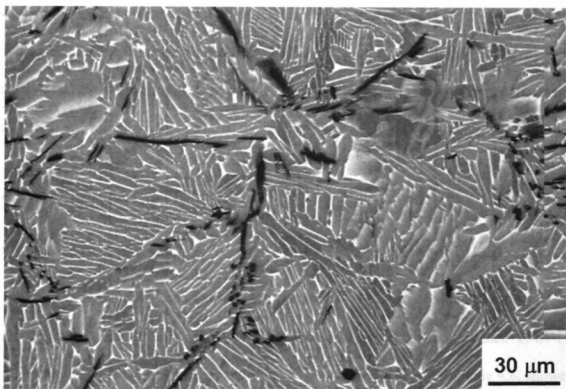


(a)

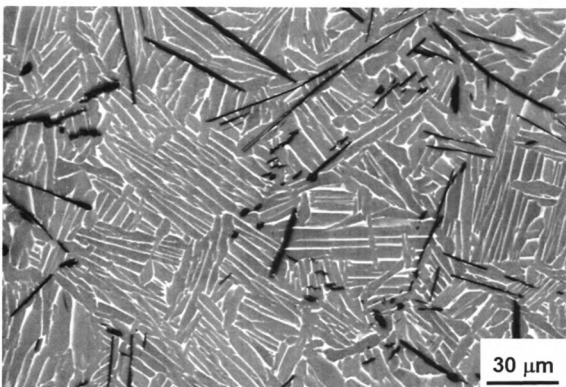


(b)

Figure 4.4 (continued)



(c)



(d)

As illustrated in Figure 4.4a-d, the effect of B addition on alloy microstructure was similar to that on the as-cast Ti-64 alloy. The volume percents of the β phase in the as-cast Ti-6242S-xB alloys ranged between 20-23%, which was slightly higher than in the as-cast Ti-64-xB alloy system. In addition, both the α - and β - lath widths of this alloy system were finer than those of the as-cast Ti-64-xB alloy system.

3. Microprobe Analysis

3.1 Ti-64-xB Alloy System

In order to determine the weight percentages of Ti, Al, V, and B in the TiB, α , and β phases, microprobe analysis was conducted on mounted and polished Ti-64-1B alloy samples of each processing condition. The microprobe data, see Table 4.6, showed that the β -phase was enriched with V, and the α -phase was enriched with Al and Ti. There was not any detectable amount of B in either the α or β phases and the TiB whiskers exhibited slightly more B than Ti when compared on the atomic percent basis. There was approximately 3.1-3.4 wt.% V (1.8-2.0 at.%) in the TiB phase.

Table 4.6. Phase Compositions in wt.% for the Ti-64-1B Alloys

Alloy	Processing	Phase	Ti*	Al*	V*	B*
Ti-64-1B	PM rolled	TiB	78.1 $\pm$ 0.7 (47.7 $\pm$ 1.0)	0.2 $\pm$ 0.2 (0.2 $\pm$ 0.2)	3.2 $\pm$ 0.1 (1.8 $\pm$ 0.1)	18.5 $\pm$ 0.6 (50.2 $\pm$ 1.0)
		α	89.4 $\pm$ 0.1 (84.4 $\pm$ 0.1)	7.9 $\pm$ 0.04 (13.2 $\pm$ 0.1)	2.7 $\pm$ 0.1 (2.4 $\pm$ 0.1)	0 (0)
		β	78.2 $\pm$ 0.4 (76.9 $\pm$ 0.3)	3.5 $\pm$ 0.1 (6.2 $\pm$ 0.2)	18.3 $\pm$ 0.5 (16.9 $\pm$ 0.5)	0 (0)
Ti-64-1B	PM extruded	TiB	77.4 $\pm$ 0.2 (46.5 $\pm$ 0.3)	0.1 $\pm$ 0.1 (0.1 $\pm$ 0.1)	3.1 $\pm$ 0.1 (1.8 $\pm$ 0.1)	19.4 $\pm$ 0.2 (51.7 $\pm$ 0.2)
		α	89.5 $\pm$ 0.2 (84.6 $\pm$ 0.2)	7.6 $\pm$ 0.2 (12.8 $\pm$ 0.2)	2.8 $\pm$ 0.3 (2.6 $\pm$ 0.3)	0 (0)
		β	81.8 (79.6)	4.7 (8.1)	13.5 (12.3)	0 (0)
Ti-64-1B	as-cast	TiB	77.8 $\pm$ 0.5 (47.4 $\pm$ 0.6)	0.06 $\pm$ 0.05 (0.1 $\pm$ 0.1)	3.4 $\pm$ 0.1 (1.9 $\pm$ 0.04)	18.7 $\pm$ 0.4 (50.4 $\pm$ 0.7)
		α	89.2 $\pm$ 0.2 (84.4 $\pm$ 0.6)	8.0 $\pm$ 0.1 (12.8 $\pm$ 1.1)	2.9 $\pm$ 0.06 (2.8 $\pm$ 0.5)	0 (0)
		β	76.9 $\pm$ 0.7 (75.9 $\pm$ 0.5)	3.2 $\pm$ 0.2 (5.6 $\pm$ 0.4)	19.9 $\pm$ 0.9 (18.5 $\pm$ 0.9)	0 (0)
Ti-64-1B	cast-and-extruded	TiB	78.3 $\pm$ 0.5 (48.5 $\pm$ 1.5)	0.1 $\pm$ 0.1 (0.1 $\pm$ 0.1)	3.5 $\pm$ 0.2 (2.0 $\pm$ 0.2)	17.9 $\pm$ 1.2 (49.1 $\pm$ 2.2)
		α	88.7 $\pm$ 0.4 (84.2 $\pm$ 0.3)	7.2 $\pm$ 0.3 (12.2 $\pm$ 0.4)	4.2 $\pm$ 0.7 (3.7 $\pm$ 0.6)	0 (0)
		β	84.2 (81.2)	5.5 (9.4)	10.4 (9.4)	0 (0)

* at.% is given in parenthesis

3.2 As-cast Ti-6242S-xB Alloy System

Microprobe analysis was conducted on mounted and polished as-cast Ti-6242S-0.4B and Ti-6242S-1B alloy samples. The microprobe data, see Table 4.7, showed that the β -phase was enriched with Mo, Zr and Sn, and the α -phase was enriched with Al and Ti. There was no detectable amount of B in either the α or β phases, and the TiB whiskers exhibited slightly more B than Ti when compared on an atomic percent basis. Roughly 1 wt.% of both Zr and Mo was measured in the TiB phase.

Table 4.7. Phase Compositions in wt.% for the Ti-6242S-0.4B and Ti-6242S-1B Alloys

Alloy	Phase	Ti	Al	Sn	Zr	Mo	Si	B
Ti-6242S-0.4B	TiB	78.1±0.3 (47.0±0.2)	0.02±0.01 (0)	0 (0)	1.0±0.2 (0.3±0.1)	1.2±0.1 (0.4)	0 (0)	19.6±0.1 (52.3±0.2)
	α	85.5±0.7 (83.2±0.2)	7.8±0.04 (13.5±0.1)	2.3±0.03 (0.9)	3.9±0.1 (2.0±0.1)	0.4±0.01 (0.2)	0.1 (0.2)	0 (0)
	β	74.6±0.5 (81.4±0.1)	3.4±0.4 (6.6±0.7)	2.9±0.1 (1.3±0.1)	5.7±0.5 (3.3±0.3)	13.3±2.4 (7.2±1.4)	0.2±0.1 (0.3±0.3)	0 (0)
Ti-6242S-1B	TiB	78.9±0.3 (48.2±0.3)	0.1±0.1 (0.1±0.1)	0 (0)	0.9±0.3 (0.3±0.1)	1.3±0.2 (0.4±0.1)	0 (0)	18.8±0.2 (50.9±0.2)
	α	85.5±0.5 (83.4±0.2)	7.6±0.2 (13.2±0.3)	2.3 (0.9)	4.1±0.2 (2.1±0.1)	0.4±0.1 (0.2)	0.1 (0.2)	0 (0)
	β	74.2±1.7 (80.9±0.7)	3.7±0.5 (7.1±0.9)	2.9 (1.3)	5.2±0.5 (3.0±0.3)	13.8±2.8 (7.5±1.7)	0.2±0.1 (0.3±0.3)	0 (0)

*at.% is given in parenthesis

4. Texture Determination

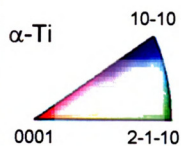
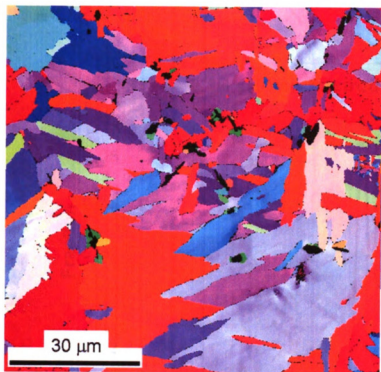
4.1 EBSD Analysis

4.1.1 As-cast and Cast-and-extruded Ti-64-xB Alloys

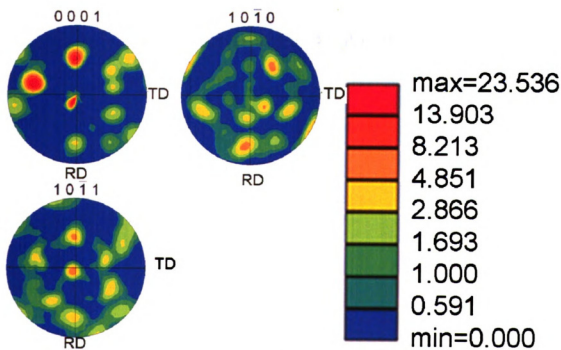
EBSD scan as performed on the transverse orientation of the sample. Although this technique was not considered to be a bulk texture measurement technique, the results were consistent with the XRD measurement as will be described next.

EBSD analysis indicated that the α and TiB phases for the cast-and-extruded alloys were strongly textured, see Figure 4.5 (cast-and-extruded Ti-64-0.1B), Figure 4.6 (cast-and-extruded Ti-64, only Ti α -phase was analyzed) and Figure 4.7 (cast-and-extruded Ti-64-1B, only Ti α -phase was analyzed). For the Ti-64-0.1B cast-and-extruded alloy, the greatest α -phase texture was more than 20 times random, while the TiB-phase texture was more than 17 times random. For the cast-and-extruded Ti-64 and Ti-64-1B alloys, the greatest α -phase texture was more than 18 and 14 times random, respectively. The TiB was textured with [020] parallel to the extrusion axis for the Ti-64-0.1B and Ti-64-1B cast-and-extruded alloys. The α -phase misorientation angle plots (see Figures 4.5c, 4.6c and 4.7c) exhibit three peaks at 0° , 60° , and 90° . For the as-cast Ti-64-xB alloys, neither the α -phase nor the TiB phase was strongly textured (see Figure 4.8 for the EBSD data of the as-cast Ti-64-1B alloy).

Figure 4.5. EBSD data from a representative scan of the cross-section (the orientation out of the page was the extrusion direction.) for a Ti-64-0.1B cast-and-extruded sample: (a) α -phase orientation map, (b) (0001), (1010), and (1011) pole figure for the α -phase, (c) α -phase misorientation angle plot, (d) TiB phase orientation map and (e) (001), (010), and (001) pole figures for the orthorhombic B27 TiB phase.

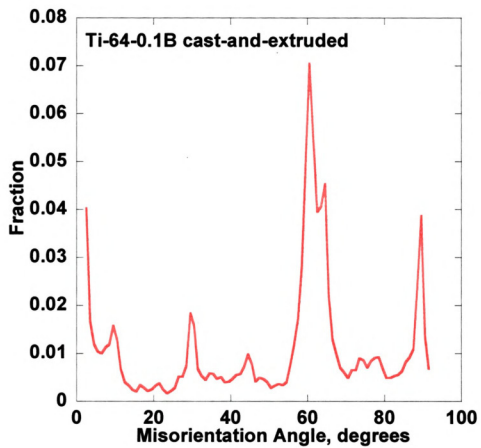


(a)



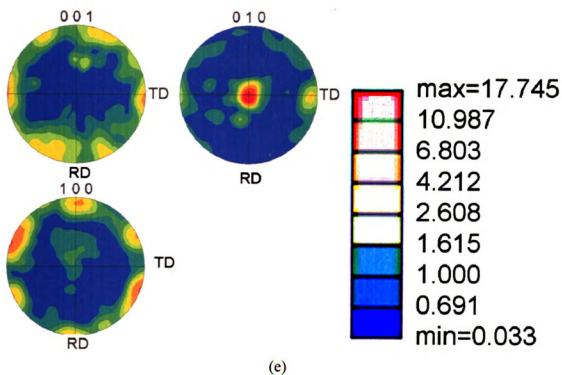
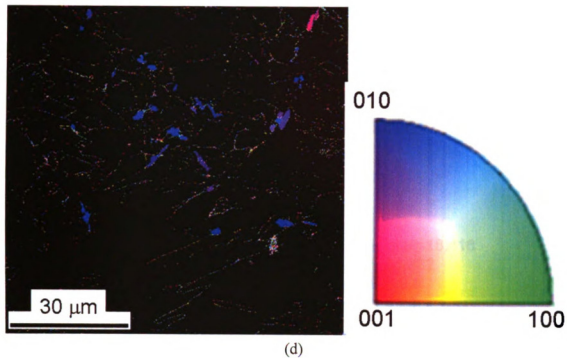
(b)

Figure 4.5 (continued)



(c)

Figure 4.5 (continued)



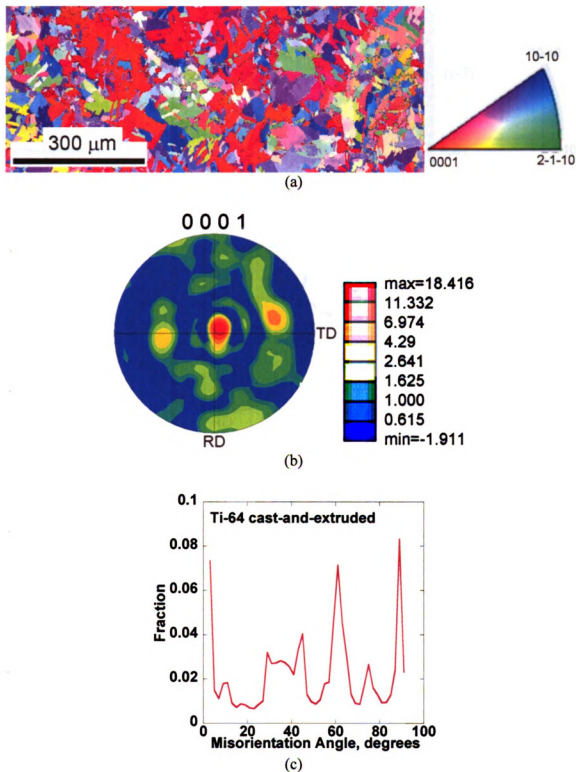


Figure 4.6. EBSD data from a representative scan of the cross-section (the orientation out of the page was the extrusion direction.) for a Ti-64 cast-and-extruded sample: (a) α -phase orientation map, (b) α -phase (0001) pole figure, and (c) α -phase misorientation angle plot. In this scan, only the Ti α -phase was analyzed.

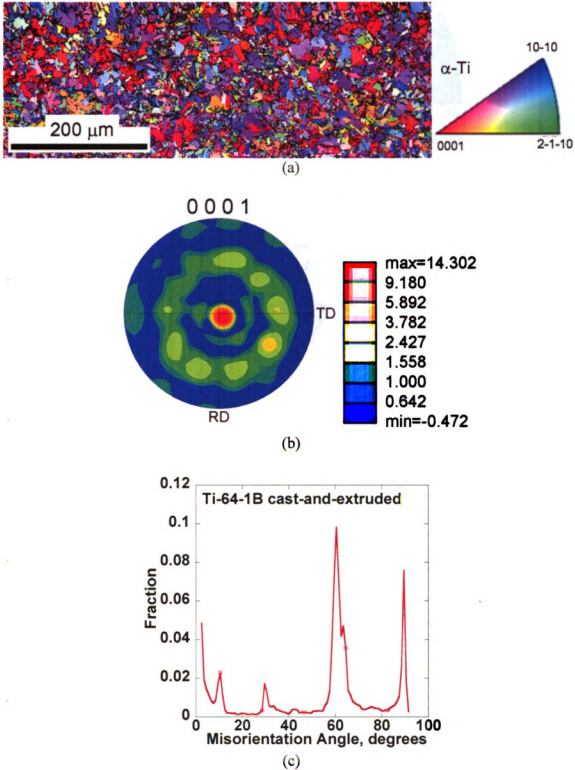


Figure 4.7. EBSD data from a representative scan of the cross-section (the orientation out of the page was the extrusion direction.) for a Ti-64-1B cast-and-extruded sample: (a) α -phase orientation map, (b) α -phase (0001) pole figure, and (c) α -phase misorientation angle plot. In this scan, only the Ti α -phase was analyzed.

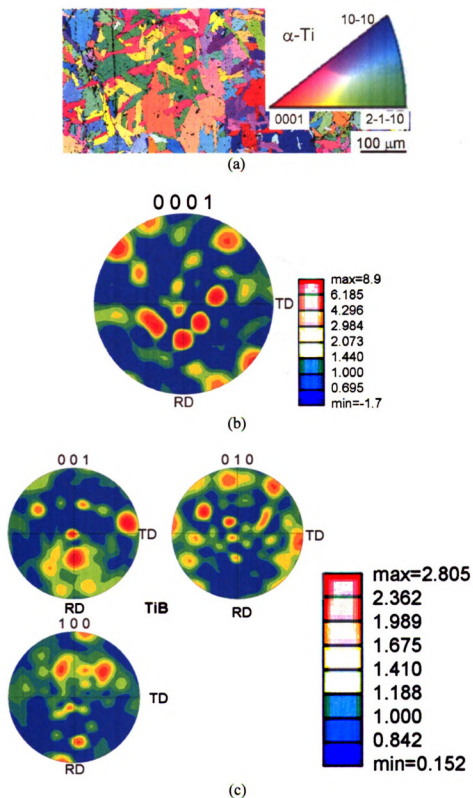


Figure 4.8. EBSD data from a representative scan of the cross-section for a Ti-64-1B as-cast sample: (a) α -phase orientation map, (b) α -phase (0001) pole figure, and (c) (001), (010), and (001) pole figures for the orthorhombic B27 TiB phase.

4.1.2 PM Processed Ti-64-1B Alloys

EBSD analysis indicated that the α and TiB phases for the PM extruded Ti-64-1B alloy were also strongly textured, see Figure 4.9. The greatest α -phase and TiB-phase texture was about four and five times random, respectively. The TiB phase was textured with [020] parallel to the extrusion axis for the PM extruded Ti-64-1B alloy. The α -phase misorientation angle plot (see Figure 4.9d) exhibited three peaks at 0° , 30° , 60° , and 90° . These peaks were not as sharp as in the cast-and-extruded Ti-64-xB alloys. The EBSD data for the PM rolled Ti-64-1B alloy is shown in Figure 4.10. The α -phase was not strongly textured (Figure 4.10b) and the misorientation angle plot shows the highest peak at 60° (Figure 4.10c). Figures 4.10d and e indicates that the TiB phase was also not strongly textured either.

Figure 4.9. EBSD data from a representative scan of the cross-section (the orientation out of the page was the extrusion direction.) for a Ti-64-1B PM extruded sample: (a) α -phase orientation map, (b) α -phase (0001) pole figure, and (c) (001), (010), and (001) pole figures for the orthorhombic B27 TiB phase, and (d) α -phase misorientation angle plot.

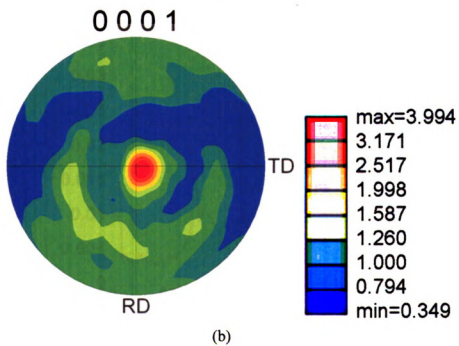
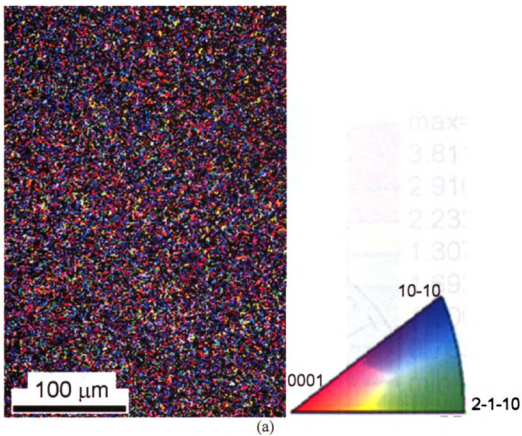
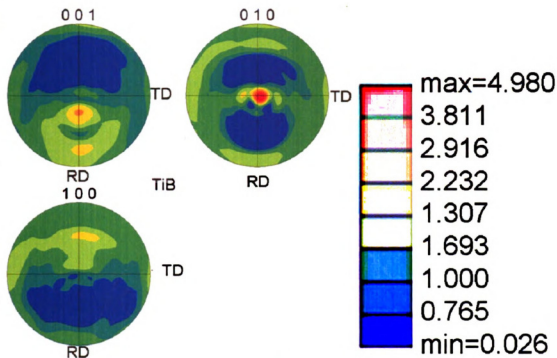
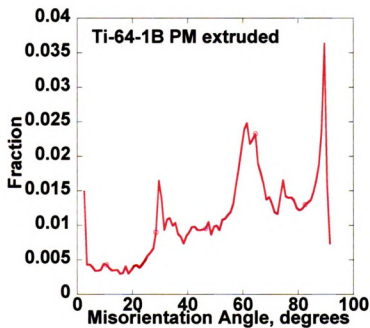


Figure 4.9 (continued)



(c)



(d)

Figure 4.10. EBSD data from a representative scan of the cross-section (the orientation out of the page was the rolling direction.) for a Ti-64-1B PM rolled sample: (a) α -phase orientation map, (b) α -phase (0001) pole figure, (c) α -phase misorientation angle plot, (d) TiB phase orientation map and (e) (001), (010), and (001) pole figures for the orthorhombic B27 TiB phase.

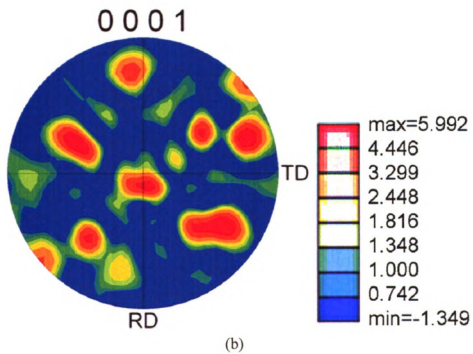
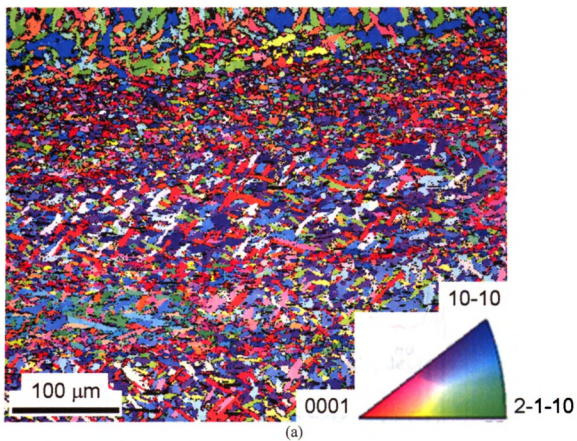


Figure 4.10 (continued)

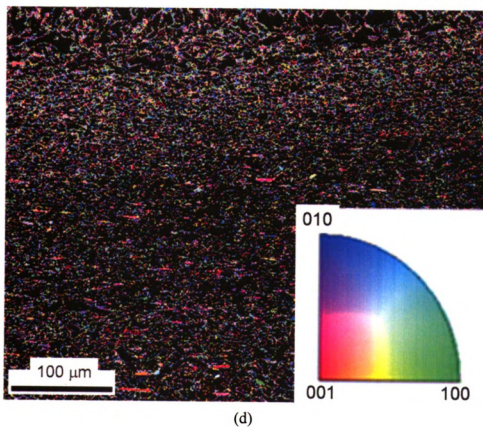
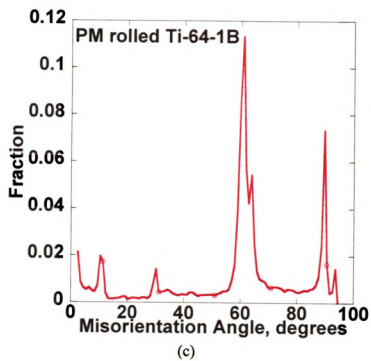
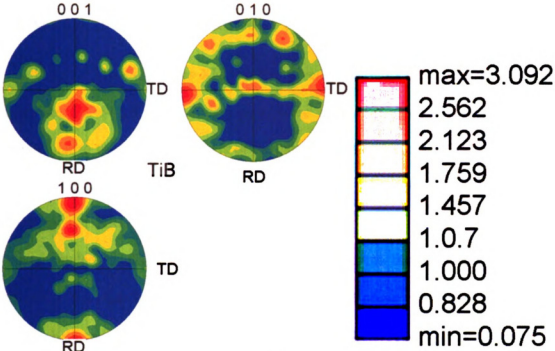


Figure 4.10 (continued)



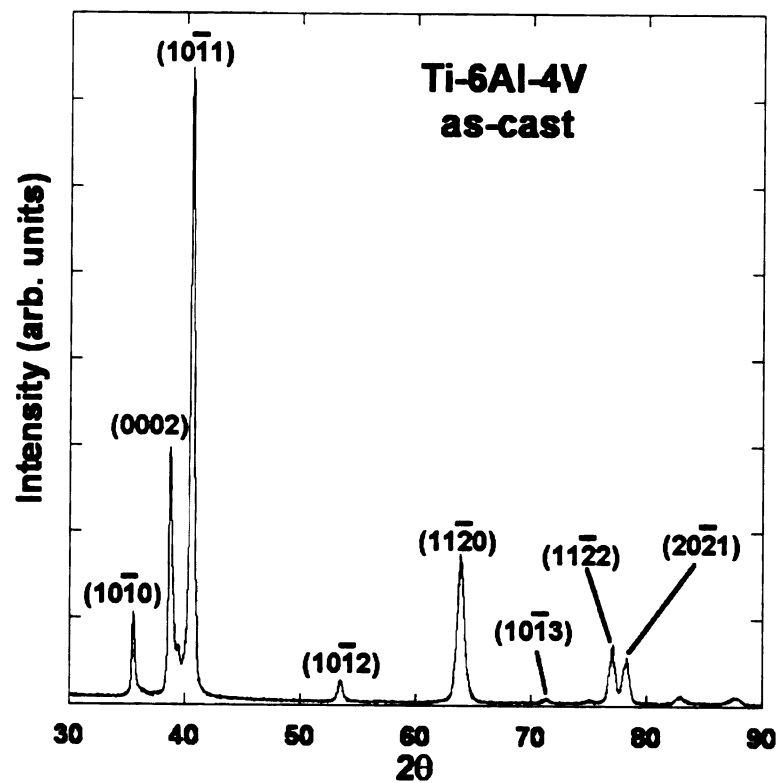
(e)

4.2 XRD Analysis

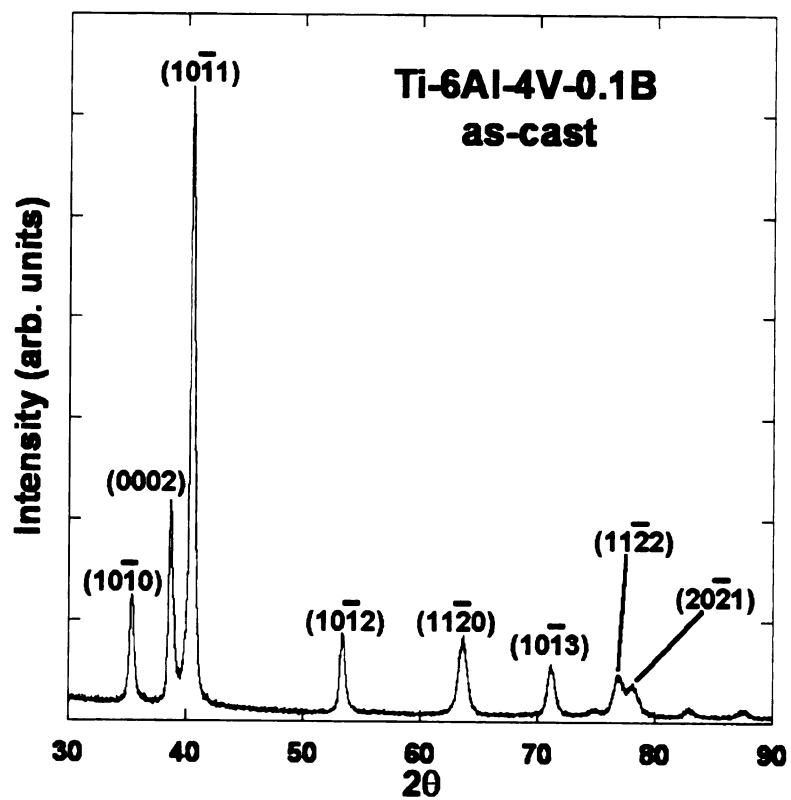
4.2.1 As-cast and Cast-and-extruded Ti-64-xB Alloys

Figure 4.11a-f illustrates XRD Intensity versus 2θ plots for the as-cast and cast-and-extruded Ti-64-xB alloys. The plots were acquired for transverse sections cut perpendicular to the longitudinal axis of the castings and extrusions. The greatest peaks for the cast-and-extruded materials were for the basal plane (0002). This has been verified by the XRD pole figures about the (0002) plane (Figure 4.12). Thus the extrusion tended to align the basal plane perpendicular to the extrusion axis. It is important to note that the largest peak for the retained β phase was for the (110) plane. Thus it is believed that the extrusion procedure resulted in a (110) β -phase texture. That is, the (110) β plane was preferentially oriented perpendicular to the extrusion direction.

Figures 4.11. XRD Intensity versus 2θ plots for transverse sections of (a) Ti-64 as-cast, (b) Ti-64-0.1B as-cast, (c) Ti-64-1B as-cast, (d) Ti-64 cast-and-extruded, (e) Ti-64-0.1B cast-and-extruded, and (f) Ti-64-1B cast-and-extruded alloys. (Measurement and characterization was assisted by Dr. Andrew Payzant of ORNL)

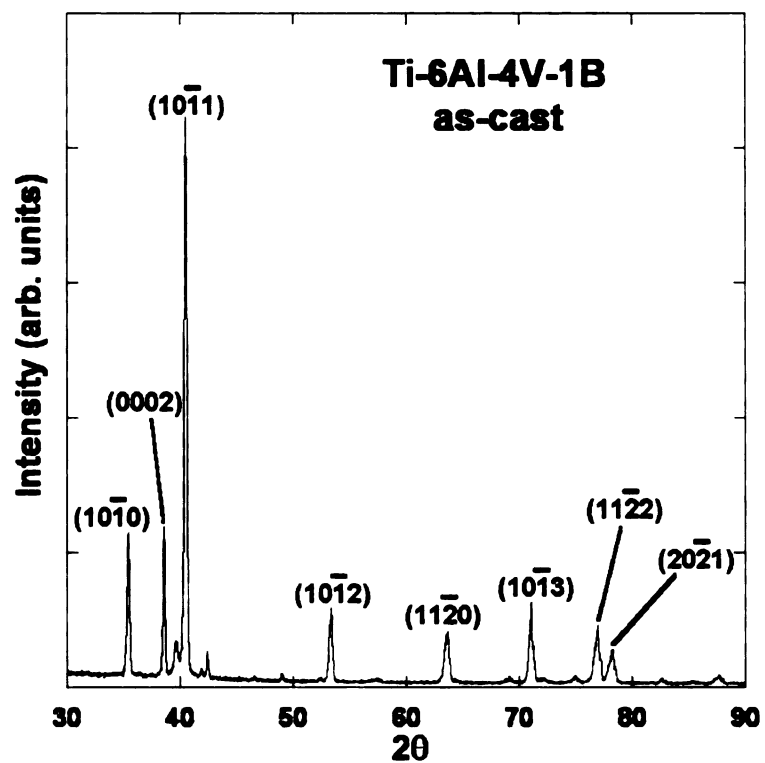


(a)

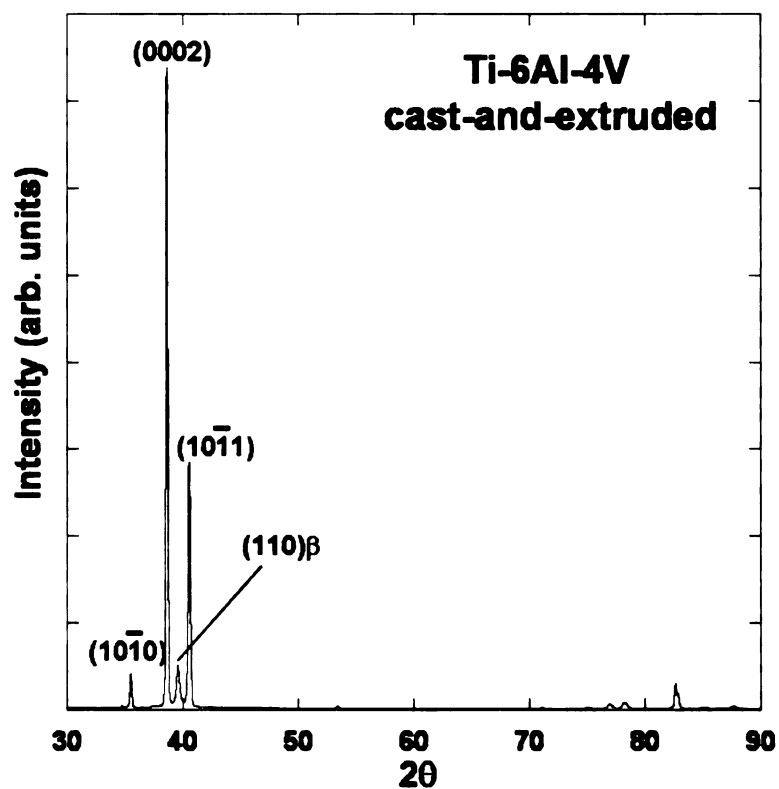


(b)

Figure 4.11 (continued)

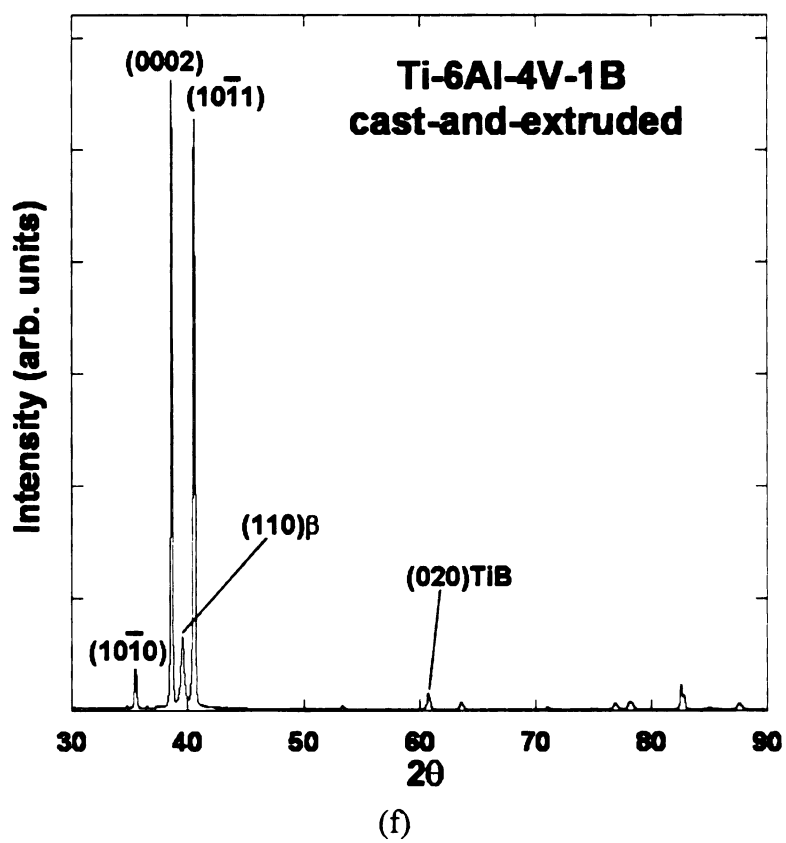
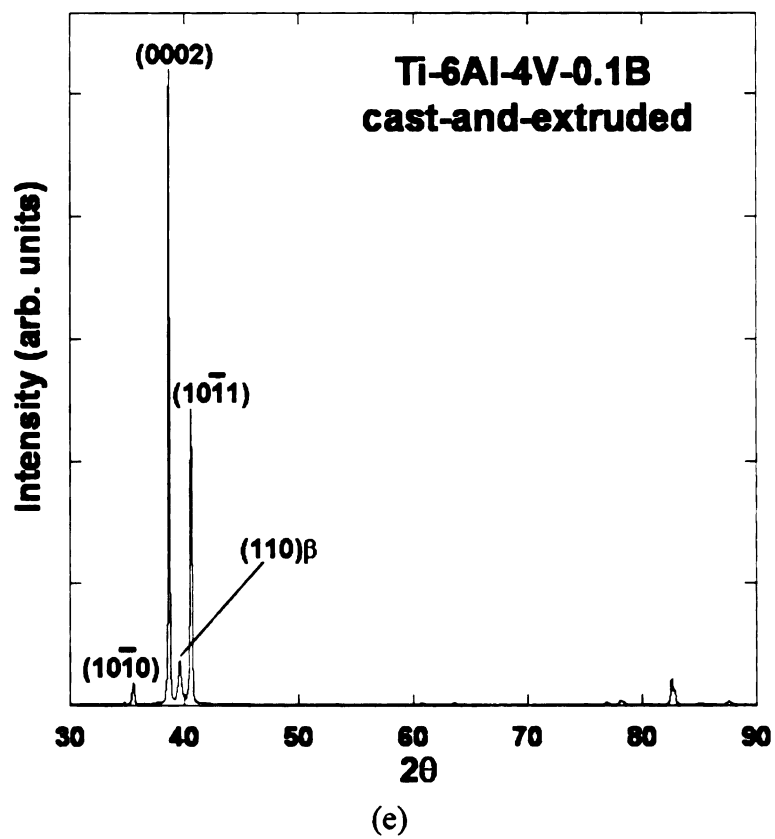


(c)



(d)

Figure 4.11 (continued)



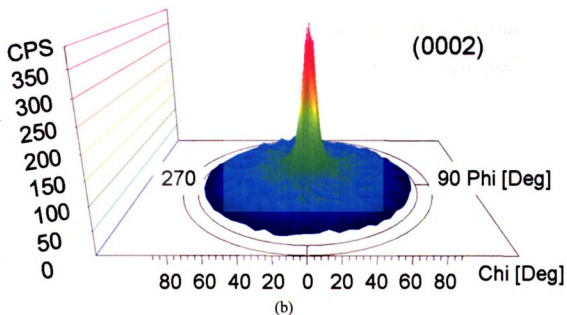
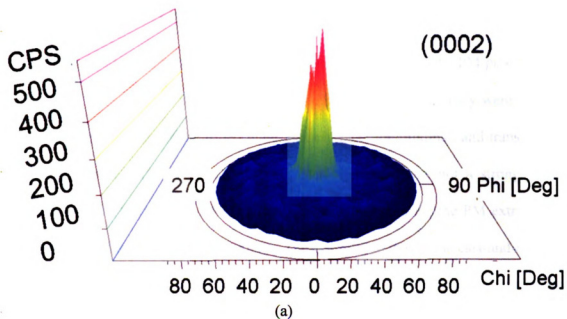


Figure 4.12. (0002) XRD Pole figure plots for the cast-and-extruded (a) Ti-64, and (b) Ti-64-1B alloys. The analysis was done on a cross section (transverse) which was cut perpendicular to the extrusion direction.

4.2.2 PM Processed Ti-64-1B Alloys

Figure 4.13a-d illustrates XRD Intensity versus 2θ plots for the PM processed Ti-64-1B alloys. The plots for the PM rolled Ti-64-1B (Figure 4.13a-c) alloy were acquired for the three orientations of the rolled plate, namely face, longitudinal, and transverse as depicted in Figure 3.10. The analysis suggested that there was not a strong texture developed in the PM rolled Ti-64-1B alloy. The greatest peak for the PM extruded Ti-64-1B alloy was for the basal plane (0002). Thus, similar to that in the cast-and-extruded Ti-64-1B alloy, the extrusion aligned the basal plane perpendicular to the extrusion axis for the PM extruded Ti-64-1B alloy. The largest peak for the retained β phase was the (110) plane. Thus it is believed that the extrusion procedure resulted in a (110) β -phase texture (i.e. (110) was perpendicular to the extrusion axis) in the PM extruded Ti-64-1B alloy.

Figures 4.13. XRD Intensity versus 2θ plots for PM processed Ti-64-1B alloys. (a) PM rolled face orientation, (b) PM rolled transverse orientation, (c) PM rolled longitudinal orientation, and (d) PM extruded transverse orientation. Note the ratio of (0002) peak and (10-11) peak in (d) compared to that for (a) (b) and (c). This indicated that the PM extruded Ti-64-1B alloy was strongly textured with (0002) perpendicular to the extrusion axis while the PM rolled alloy was not strongly textured.

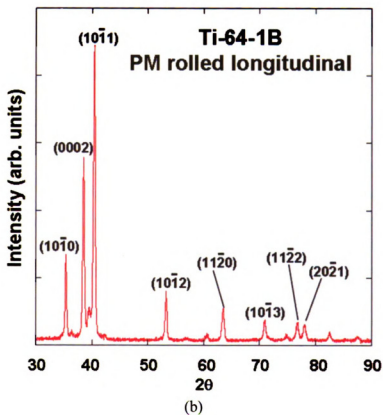
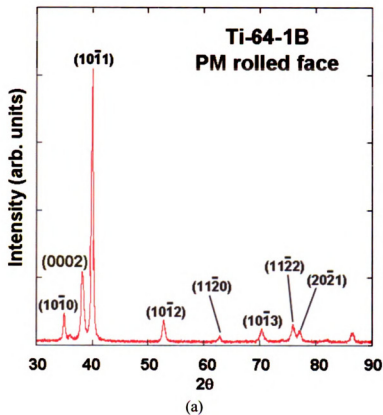
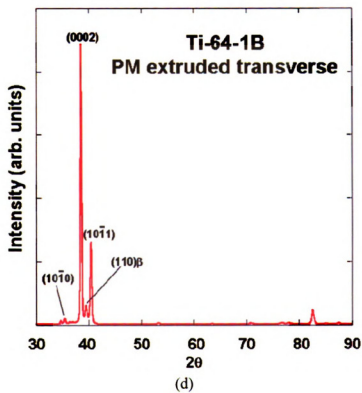
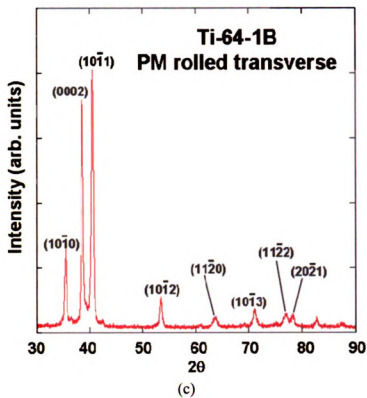


Figure 4.13 (continued)



B. Tensile Behavior Results

1. Ti-64-xB Alloy System

1.1 As-cast and Cast-and-extruded Alloys

1.1.1 Tensile Properties

Each of the samples was tested in the direction parallel to the casting, and/or extrusion directions (see Figure 3.11 in Experimental section for reference). The 455°C tensile properties for the studied alloys are listed in Table 4.8, and Figure 4.14 illustrates the 455°C tensile stress versus strain behavior of representative samples. The YS and UTS values of the as-cast alloys were significantly lower than the cast-and-extruded alloys. Greater B contents resulted in greater tensile strengths for the as-cast alloys, but the strengths of the cast-and-extruded alloys were relatively insensitive to B content. The as-cast Ti-64-1B alloy exhibited the lowest ϵ_f (2.5%) value. The ϵ_f value for the Ti-64-1B cast-and-extruded alloy was 8.2%. Each of the other alloys exhibited a minimum ϵ_f value of 9.7%. Table 4.9 lists the RT tensile properties for the Ti-64-xB alloys (the tensile properties for the as-cast Ti-64-xB alloys were taken from Sen *et al.* [Sen (2007)] and Tamirisakandala *et al.* [Tamiriskandala (2005)]). Similar to that at 455°C, the strengths of the cast-and-extruded alloys were insensitive to B content at RT. However, the RT tensile strengths and E values were significantly higher than those at 455°C.

Table 4.8. Average 455°C Tensile Properties for the Ti-64-xB As-cast and Cast-and-extruded Alloys

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64	as-cast	90	379	435	9.7
Ti-64-0.1B	as-cast	94	435	552	11.8
Ti-64-1B	as-cast	103	513	614	2.5
Ti-64	cast-and-extruded	102	676	750	10.7
Ti-64-0.1B	cast-and-extruded	105	640	747	10.8
Ti-64-1B	cast-and-extruded	113	663	785	8.2

*Each of the samples was tested in the direction parallel to the casting, and/or extrusion directions (see Figure 3.11 in Experimental section for reference).

Table 4.9. Room Temperature (22°C) Tensile Properties for the Ti-64-xB As-cast and Cast-and-extruded Alloys

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64*	as-cast	107	834	900	6.0
Ti-64-0.1B*	as-cast	116	900	966	7.2
Ti-64-1B**	as-cast	146	975	1045	2.5
Ti-64	cast-and-extruded	125	1033	1095	10.5
Ti-64-0.1B	cast-and-extruded	126	1066	1083	7.2
Ti-64-1B	cast-and-extruded	128	1043	1121	6.1

*[Sen (2007)], **[Tamirisakandala (2006)]

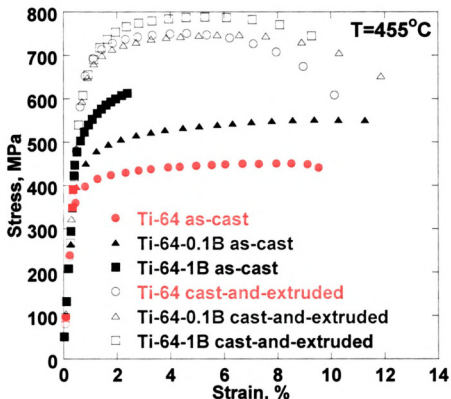


Figure 4.14. The 455°C tensile stress versus strain behavior of the studied alloys.

As has been described previously in this chapter, the cast-and-extruded Ti-64-xB alloys were strongly textured with the basal plane of α phase perpendicular to the extrusion axis. Tensile tests in the transverse direction of the extrusion bars were also performed (see Figure 3.14 in Experimental section for orientation of sample with respect to extrusion axis). These tests were performed to assess the effect of texture on the tensile properties of the cast-and-extruded Ti-64-xB alloys. The SEM image in Figure 4.15a shows the setup of a representative sample at 455°C and Figure 4.15b shows a fractured sample (Ti-64-xB cast-and-extruded alloy) after the tensile test. The sample broke in the middle of the gage section at 45° with respect to the load direction, which suggests a ductile failure.

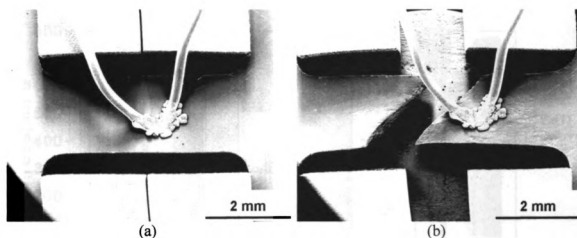
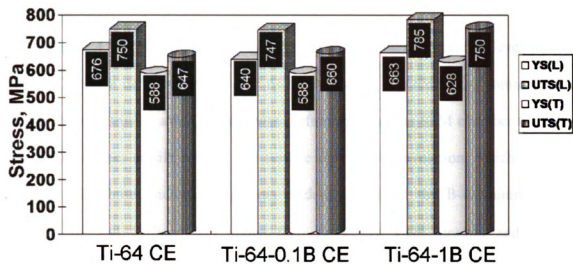
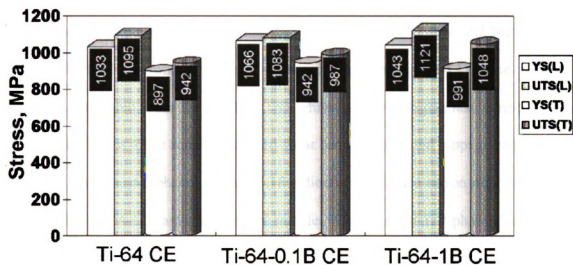


Figure 4.15. SE SEM images of tensile test for a cast-and-extruded Ti-64-xB sample tested perpendicular to the extrusion axis at 455°C. (a) before the load was applied, and (b) after the broken of the sample. A type K thermocouple was spot welded to the surface of the sample to measure the temperature, which was altered using a resistive heater located underneath the sample.

The YS and UTS in both the longitudinal (L) and transverse (T) directions are shown in Figures 4.16a (455°C) and 4.16b (RT). It is clear that the tensile strength in the longitudinal direction for all the Ti-64-xB cast-and-extruded alloys is higher than that in the transverse direction. This anisotropy with respect to the tensile properties resulted from the texture caused by the extrusion process. Notice that the tensile anisotropy is more pronounced for the cast-and-extruded Ti-64 and Ti-64-0.1B alloys. The UTS difference between longitudinal and transverse directions was 103 MPa at 455°C and 153 MPa at RT for cast-and-extruded Ti-64 alloy. For the cast-and-extruded Ti-64-0.1B alloy, the difference was 87 MPa at 455°C and 96 MPa at RT. However, the UTS difference for the cast-and-extruded Ti-64-1B alloy was smaller (35 MPa at 455°C and 73 MPa at RT).



(a)



(b)

Figure 4.16. Comparison of YS and UTS in longitudinal (L) direction (plotted with rectangular columns) and transverse (T) direction (plotted with cylinders) for the Ti-64-xB cast-and-extruded alloys; (a) 455°C properties and (b) RT (22°C) properties.

1.1.2 *In-situ* Tensile Experiments

Figure 4.17 illustrates images taken at sequential load/displacement levels during the 455°C tensile deformation for a Ti-64-0.1B cast-and-extruded alloy sample tested parallel to the extrusion axis. This test was performed inside an SEM chamber using the Ernest Fullam Inc. tensile stage. The surface deformation evolution, which included extensive TiB-phase microcracking, was evident. Each of the B-containing alloys exhibited α - and β -phase slip above their respective yield stresses and surface microcracks in the TiB whiskers were observed at stresses well below the global yield stress. In some cases, the TiB whisker cracks were observed at stresses more than 150 MPa below the global yield stress of the alloy. The microcracks grew and became more extensive in the TiB whiskers as the load was increased, see Figure 4.17. The α - and β -phase slip emanated from the cracked TiB whiskers where high-stress concentrations existed, see Figures 4.17e and f. Localized shear bands formed and propagated leading to cracking in the α and β phases. Figure 4.18 indicates the surface region near the fracture location for a Ti-64-1B cast-and-extruded sample. The extent of TiB-phase cracking was evident. The extent of matrix slip as well as dimples on the fracture surfaces confirmed that the B-containing cast-and-extruded alloys tolerated large elongations before failure occurred even though microcracking in the TiB whiskers occurred prior to global yielding. Features similar to those observed on the surface (multiple TiB cracking) were observed in the interior of the samples as well.

Figure 4.17. SE SEM images of the surface for a Ti-64-0.1B sample tensile tested at $T=455^{\circ}\text{C}$ (a)-(m). The loading axis was horizontal in the images. The TiB phase cracking evolution is evident. This sample was not taken to failure.

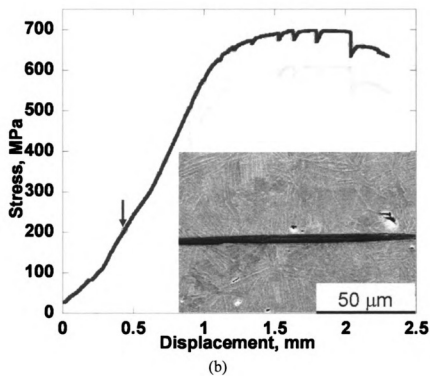
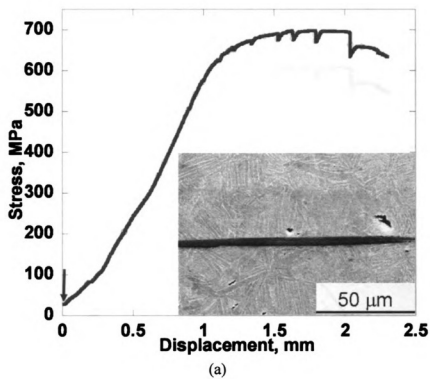


Figure 4.17 (continued)

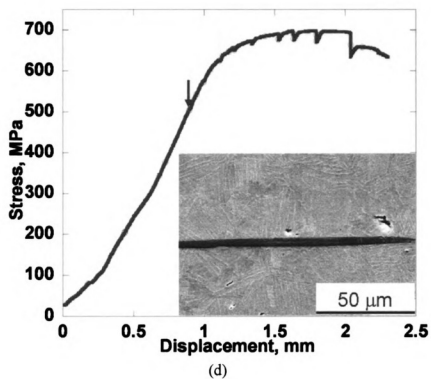
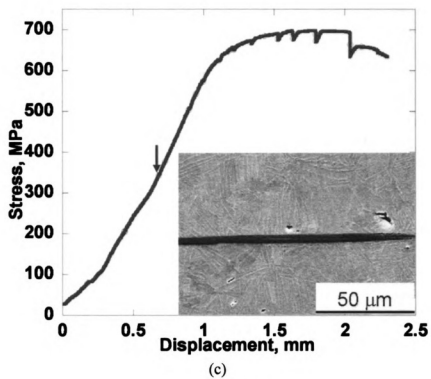


Figure 4.17 (continued)

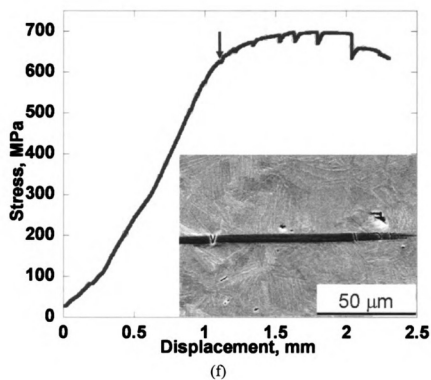
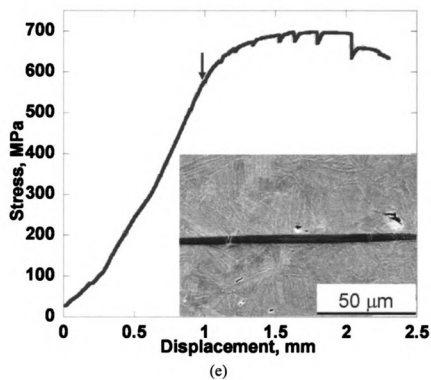


Figure 4.17 (continued)

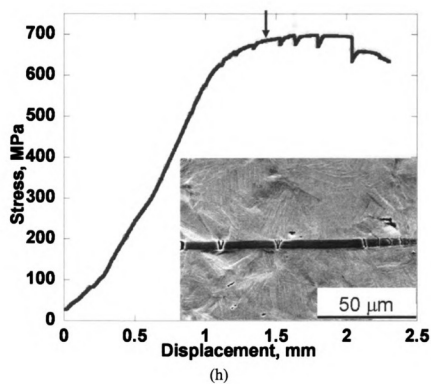
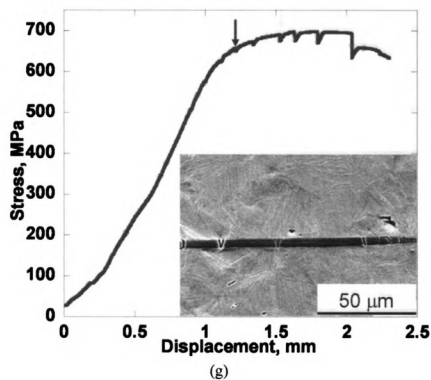


Figure 4.17 (continued)

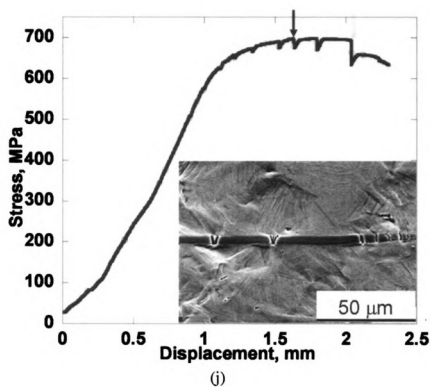
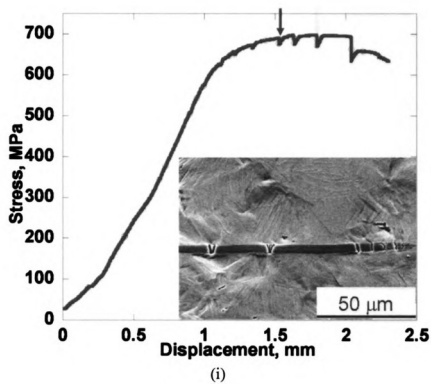


Figure 4.17 (continued)

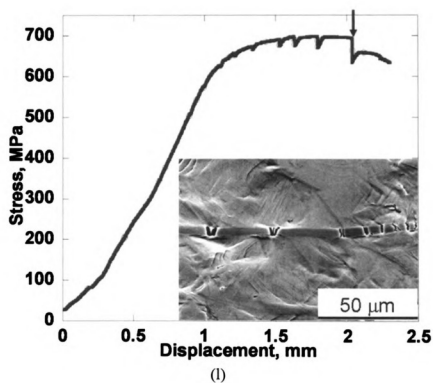
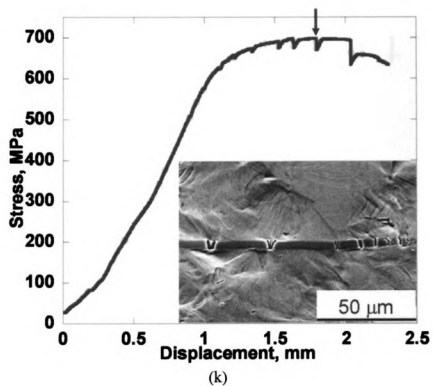
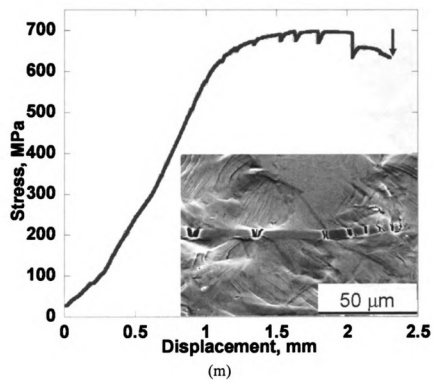


Figure 4.17 (continued)



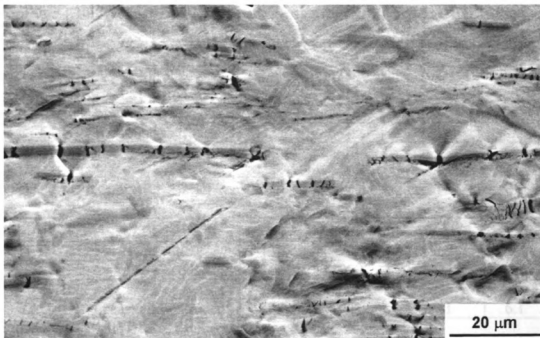


Figure 4.18. Secondary electron SEM images of the surface for a Ti-64-1B cast-and-extruded sample tensile tested at $T=455^{\circ}\text{C}$. The loading axis was horizontal and the image was taken within $100\text{ }\mu\text{m}$ of the fracture surface. The TiB phase cracking was evident.

1.2 PM Processed Ti-64-1B Alloys

The 455°C tensile properties for the PM processed Ti-64-1B alloys are listed in Table 4.10 and the RT tensile properties are listed in Table 4.11. The tensile properties of as-cast and cast-and-extruded Ti-64-1B alloys are also included for comparison. Figure 4.19 illustrates the 455°C tensile stress versus strain behavior of representative samples. The YS and UTS values of the PM rolled and as-cast Ti-64-1B alloys were significantly lower than the PM extruded and cast-and-extruded Ti-64-1B alloys. The YS of the PM rolled Ti-64-1B alloy is similar to that of the as-cast Ti-64-1B alloy, but its ϵ_f is much higher. The YS, UTS and ϵ_f of the PM extruded Ti-64-1B alloy were similar to those of the cast-and-extruded Ti-64-1B alloy.

Table 4.10. Average 455°C Tensile Properties for the Studied Ti-64-1B Alloys

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64-1B	PM rolled	100	538	702	8.7
Ti-64-1B	PM extruded	117	691	804	9.0
Ti-64-1B	as-cast	103	513	614	2.5
Ti-64-1B	cast-and-extruded	113	663	785	8.2

Table 4.11. Average RT (22°C) Tensile Properties for the Studied Ti-64-1B Alloys

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64-1B*	PM rolled	138	965	1069	10.0
Ti-64-1B**	PM extruded	142	1079	1224	13.0
Ti-64-1B**	as-cast	146	975	1045	2.5
Ti-64-1B	cast-and-extruded	128	1043	1121	6.1

*[Schwendiman (2007)], **[Tamirisakandala (2006)]

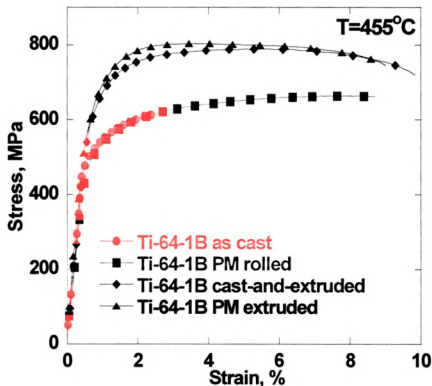


Figure 4.19. The 455°C tensile stress VS. strain behavior of representative Ti-64-1B alloys for each processing condition.

As has been shown previously in this chapter, the PM extruded Ti-64-1B alloy was strongly textured with the basal plane of the α phase perpendicular to the extrusion axis. The TiB phase was also strongly textured in the PM extruded Ti-64-1B alloy. Tensile tests in the transverse direction for the PM processed Ti-64-1B alloys were also performed in order to evaluate this anisotropy with respect to the tensile properties. Although the PM rolled Ti-64-1B alloy does not exhibit a strong texture, the tensile test was also performed and the sample was cut perpendicular to the rolled plate.

The YS and UTS in both longitudinal and transverse directions at 455°C are shown in Figure 4.20a. The RT properties are shown in Figure 4.20b. For the PM rolled Ti-64-1B alloy, the tensile properties in longitudinal and transverse directions were similar (both at 455°C and RT). This is reasonable as the PM rolled Ti-64-1B alloy was not strongly textured. However, for the PM extruded Ti-64-1B alloy, the YS and UTS in the longitudinal direction are higher than those in the transverse direction, which is similar to the cast-and-extruded Ti-64-xB alloys. This anisotropy with respect to the tensile strength for the PM extruded Ti-64-1B alloy was a result of the texture caused by the extrusion process.

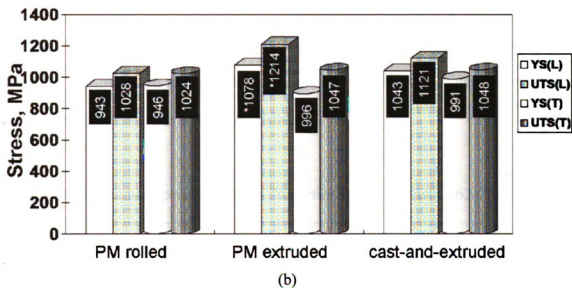
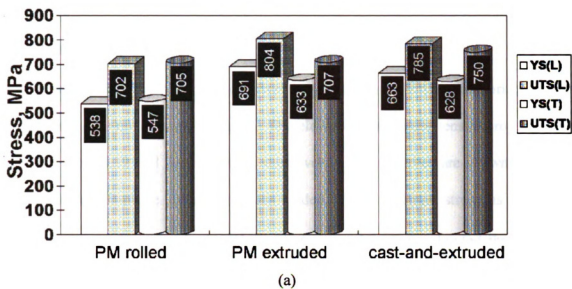


Figure 4.20. Comparison of YS and UTS in longitudinal (L) direction (plotted with columns) and transverse (T) direction (plotted with cylinders) for the Ti-64-1B alloys. (a) 455°C properties and (b) RT (22°C) properties. *[Lieberman (2007)]

2. As-cast Ti-6242S-xB Alloy System

2.1 Tensile Properties

The as-cast Ti-6242S-xB alloys were only tested in the longitudinal orientation as this material was not strongly textured. The elevated-temperature tensile properties are listed in Table 4.12 and representative stress versus strain curves are shown in Figure 4.21. Larger TiB-phase volume fractions tended to lead to larger strengths and larger Young's modulus values. At 455°C, the Ti-6242S-0.1B alloy exhibited the largest ϵ_f value (10.3%), while the Ti-6242S-0.4B and Ti-6242S-1B alloys exhibited brittle fracture and ϵ_f values of approximately 1%. In general, the alloys' yield strengths decreased with increased temperature. The stress versus displacement curve for a Ti-6242S-1B alloy tested at 565°C is shown in Figure 4.22. The various stress levels at which the test was paused for SEM imaging is evident on this plot.

Table 4.12. Elevated-temperature Tensile Properties for the As-cast Ti-6242S-xB Alloys

Composition	Temperature, °C	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-6242S	455	97	542	569	8.2
Ti-6242S-0.1B	455	95	587	660	10.3
Ti-6242S-0.4B	455	97	663	667	1.1
Ti-6242S-0.4B	510	N/A	526	669	N/A
Ti-6242S-0.4B	565	N/A	500	667	N/A
Ti-6242S-1B	455	112	660	707	1.3
Ti-6242S-1B	510	N/A	552	645	N/A
Ti-6242S-1B	565	N/A	500	652	N/A

N/A: not available

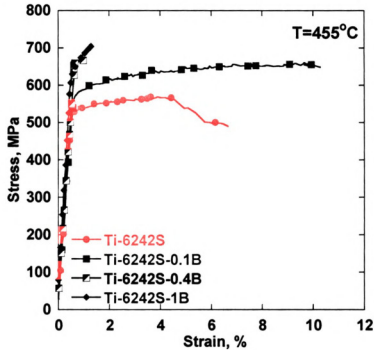


Figure 4.21. The 455°C tensile stress VS. strain plot of the as-cast Ti-6242S-xB alloys.

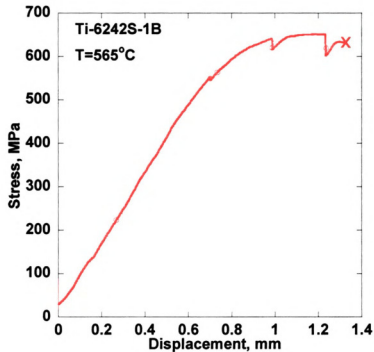
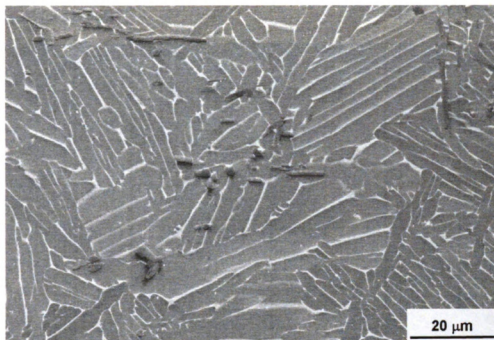


Figure 4.22. Tensile stress VS. displacement plot for a Ti-6242S-1B specimen at 565°C. The "X" indicates fracture. The test was paused at different intervals for imaging and these intervals are identified by the reduction in load that occurred and/or the open circles.

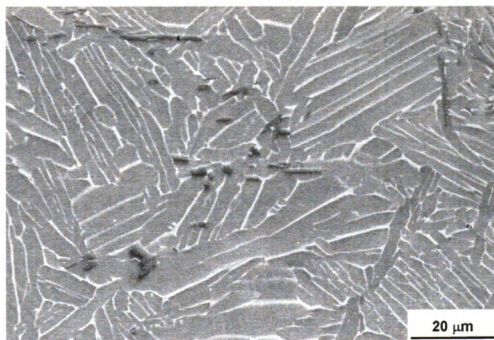
2.2 *In-situ* Tensile Tests

Figures 4.23 and 4.24 illustrate images taken at sequential load/displacement levels during the 565°C tensile deformation for the Ti-6242S-1B (see Figure 4.22 for the stress vs. strain plot) and Ti-6242S-0.4B alloy samples, respectively. Similar to the Ti-64-xB alloy system, these alloys exhibited global yielding above their respective yield stresses and surface microcracks in the TiB whiskers were observed at stresses well below the global yield stress. In some cases, the TiB whisker cracks were observed at stresses more than 100 MPa below their global yield stress. The microcracks grew and became more extensive in the TiB whiskers as the load was increased. The α - and β -phase slip emanated from the cracked TiB whiskers where high-stress concentrations existed. Localized shear bands formed and propagated leading to cracking in the α and β phases. The extent of matrix slip as well as dimples on the fracture surfaces (Figure 4.25) confirmed that the alloy tolerated some elongation before failure occurred even though microcracking in the TiB whiskers occurred prior to global yielding. The above-mentioned surface deformation observations were typical for all the elevated-temperature tensile samples tested. Features similar to those observed on the surface (multiple TiB cracking) were observed in the interior of the samples as well.

Figure 4.23. (a)-(e) SE SEM images for a Ti-6242S-1B sample tensile tested at 565°C. The loading axis was horizontal in the images. The displacements/loads were indicated. The TiB phase cracking evolution is evident in these images. Arrows indicate slip lines in the $\alpha+\beta$ microstructure.



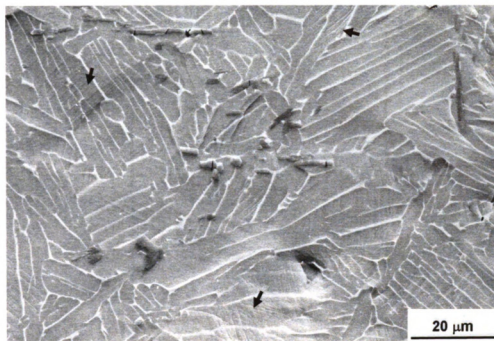
(a) 0MPa/0mm



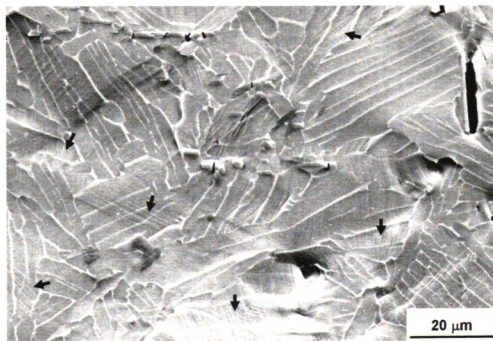
(b) 550MPa/0.704mm

ed at 565°C
re indicated
slip lines in

Figure 4.23 (continued)

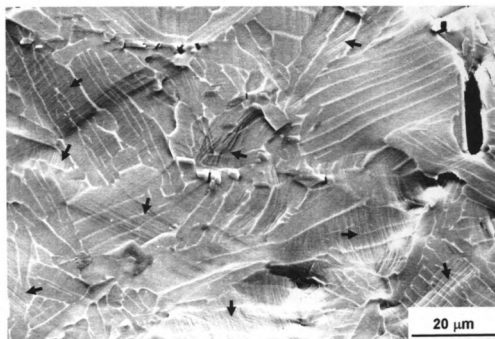


(c) 640MPa/0.988mm



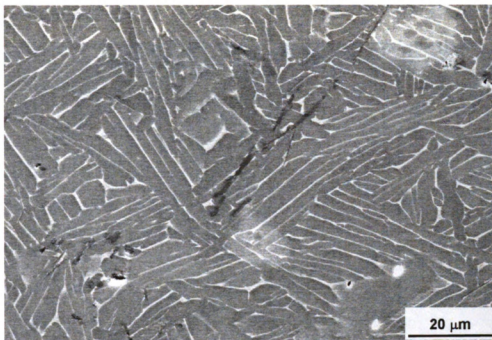
(d) 650MPa/1.232mm

Figure 4.23 (continued)

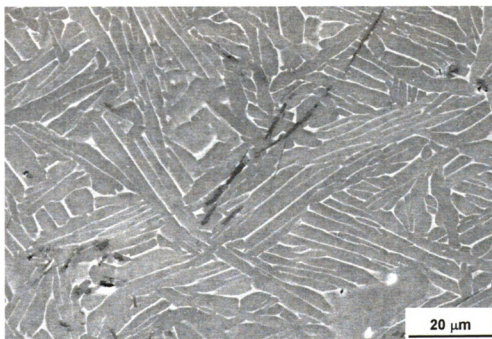


(c) post failure

Figure 4.24. (a)-(f) SE SEM images for a Ti-6242S-0.4B sample tensile tested at 565°C. The loading axis was horizontal in the images. The displacements/loads were indicated. The TiB phase cracking evolution is evident in these images. Slip lines in the $\alpha+\beta$ microstructure are evident.

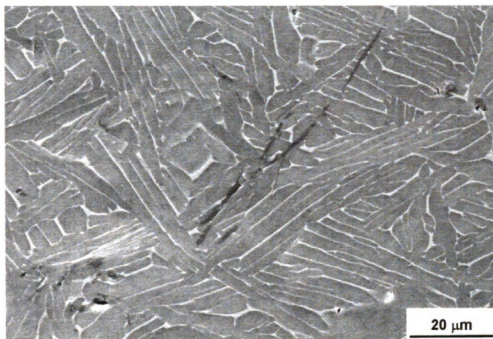


(a) 0MPa/0mm

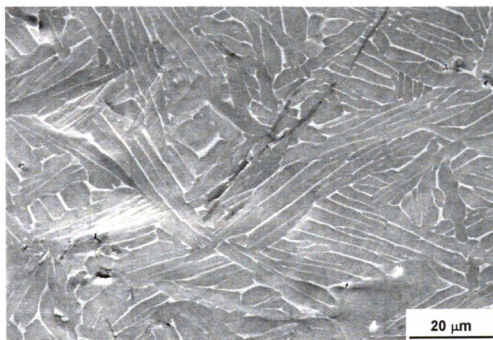


(b) 500MPa/0.833mm

Figure 4.24 (continued)

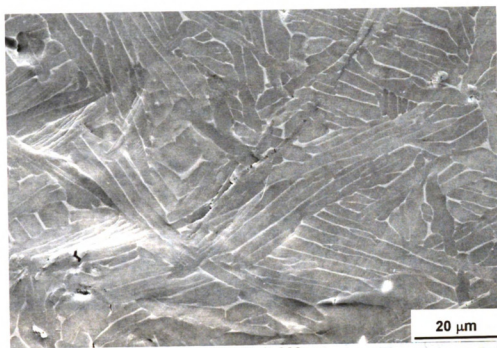


(c) 575MPa/0.960mm

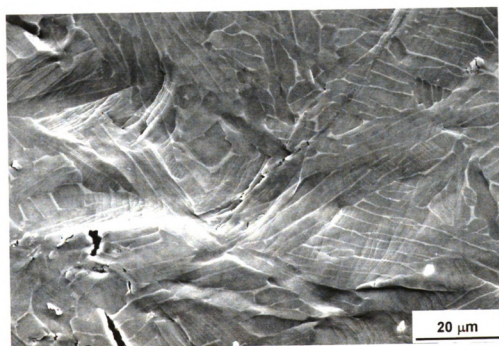


(d) 630MPa/1.128mm

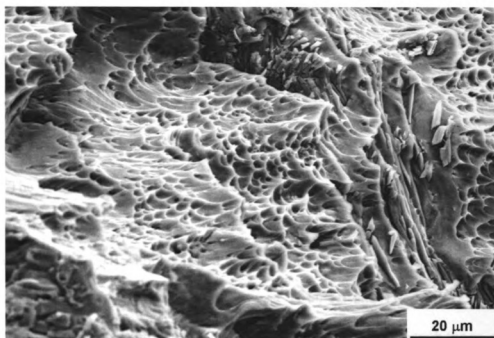
Figure 4.24 (continued)



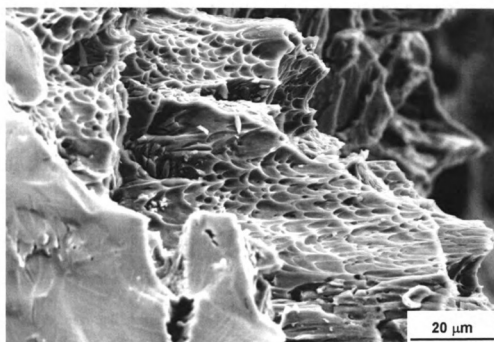
(e) 650MPa/1.288mm



(f) post failure



(a)



(b)

Figure 4.25. SE SEM image of the fracture surface of (a) Ti-6242S-1B and (b) Ti-6242S-0.4B tensile samples tensile tested at 565°C where ductile dimples were exhibited. The tensile axis was horizontal.

C. Fatigue Behavior Results

1. Ti-64-xB Alloy System

1.1 As-cast and Cast-and-extruded Alloys

1.1.1 Fatigue Behavior (455°C)

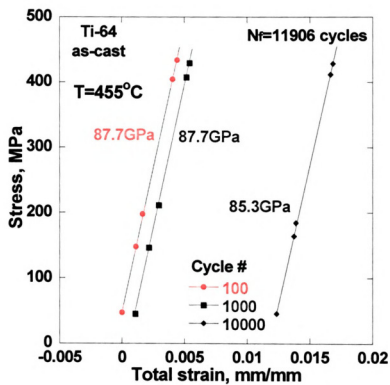
The 455°C fatigue S-N curves obtained for the as-cast Ti-64-xB alloys are shown in Figure 4.26a. For the maximum stress levels of 350 MPa, 400 MPa, and 450 MPa, the three as-cast Ti-64-0.1B samples tested each exhibited longer lives than the samples of the other as-cast alloys. Comparing the as-cast Ti-64-1B and Ti-64 alloys, there was overlap in the N_f ranges exhibited at a given maximum stress level, though the as-cast Ti-64-1B alloy exhibited greater average N_f values. It is noted that the S-N behavior for the as-cast Ti-64 alloy was quite similar to that for a cast-then-forged Ti-64 alloy, with an α -lath width size between 1-2 μm , tested at 450°C [Nalla (2003)]. The 455°C fatigue S-N curves obtained for the cast-and-extruded Ti-64-xB alloys are shown in Figure 4.26b. The fatigue lives of these alloys were superior to those for the as-cast alloys. Unlike that for the as-cast alloys, the B-modification of the cast-and-extruded alloys did not result in improved fatigue strength. In fact, comparing the cast-and-extruded alloys at the maximum applied stress of 550 MPa, the B-containing alloys always exhibited lower N_f values than the cast-and-extruded Ti-64 alloy. For the cast-then extruded alloys, the Ti-64 alloy exhibited greater average fatigue lives than the B-modified alloys.

For the as-cast fatigue samples which fractured, stress versus strain plots during defined fatigue cycles are shown in Figures 4.27a, b, and c. The E value tended to decrease with increasing cycle number indicating that damage was occurring in the samples. The damage was observed to be in the form of both TiB-phase cracking and α -phase cracking which will be shown later. An E value decrease of 14% was the maximum decrease for an as-cast Ti-64-0.1B sample tested at $\sigma_{\max}=400$ MPa (E=88 GPa at cycle 100 and E=76 GPa at cycle 900,000; $N_f=990,554$ cycles). However, usually only a 5% decrease in E was exhibited by the as-cast samples prior to fracture. For each runout sample, an E value decrease was not observed. This suggests that fatigue damage did not occur. Selected fatigue runout samples were tensile tested at 455°C, see Table 4.13. Comparing these results to those in Table 4.8, it is evident that there was no strength loss due to fatigue. This further suggests that no fatigue damage occurred to the samples that experienced runout. The cast-and-extruded samples did not show an E value decrease during the experiments, see Figures 4.27d, e, and f. The shift of the hysteresis with increasing cycle number indicates creep occurred during the fatigue tests. This increased creep is reasonable based on the 455°C creep behavior of these alloys, as will be discussed later in the results section on the creep behavior. No phase instability was observed on the post-fatigue gage sections for any of the samples tested even after more than two million cycles of fatigue exposure at 455°C. In such cases the measured volume percents of the TiB, α , and β phases were similar both before and after the experiments.

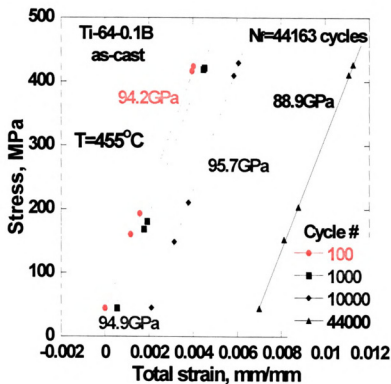
Table 4.13. Average 455°C Tensile Properties for the Runout Ti-64-xB As-cast and Cast-and-extruded Specimens

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64	as-cast	84	389	417	8.9
Ti-64-0.1B	as-cast	95	434	537	5.9
Ti-64-1B	as-cast	107	544	603	1.4
Ti-64	cast-and-extruded	97	645	771	11.8
Ti-64-0.1B	cast-and-extruded	106	677	800	9.5
Ti-64-1B	cast-and-extruded	109	676	804	8.8

Figure 4.27. Stress versus strain plots at the marked cycle numbers for the studied alloys tested at 455°C. (a) Ti-64 as-cast, (b) Ti-64-0.1B as-cast, (c) Ti-64-1B as-cast, (d) Ti-64 cast-and-extruded, (e) Ti-64-0.1B cast-and-extruded, and (f) Ti-64-1B cast-and-extruded. The slope of each curve is indicated by the listed Young's modulus value.

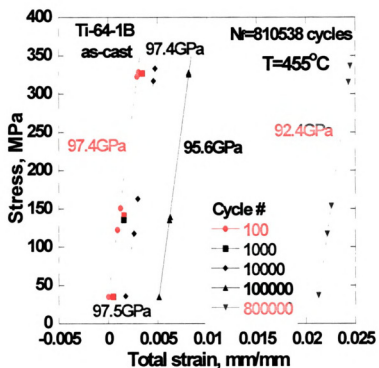


(a)

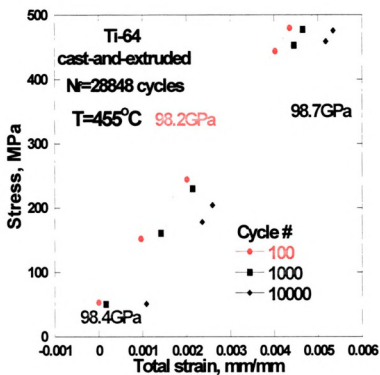


(b)

Figure 4.27 (continued)

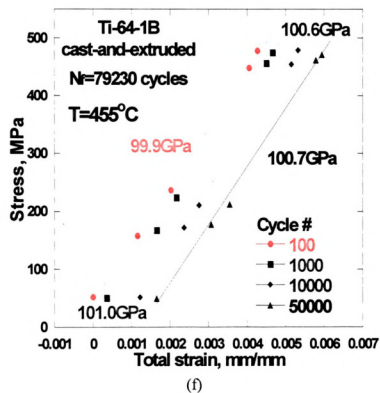
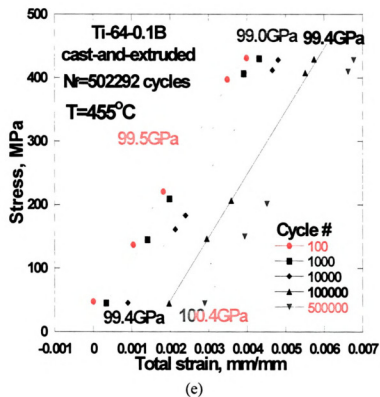


(c)



(d)

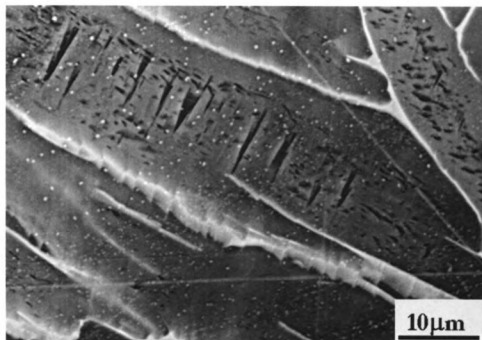
Figure 4.27 (continued)



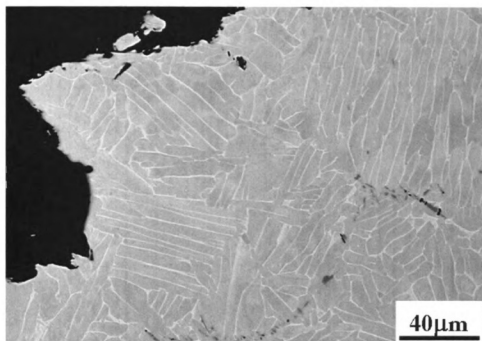
1.1.2 Fracture Analysis

For the B-containing as-cast alloys, TiB-phase cracking was evident within the deformed gage sections, see Figures 4.28b and c. This behavior was similar to that for a Ti-64/TiB alloy containing 0.5 wt.% B [Soboyejo (2004)]. The α -phase also exhibited a significant amount of cracking, see Figure 4.28a, while cracks were only rarely observed within the fine β phase. For each alloy, cracks were observed along the edges of the samples and grew perpendicular to the loading direction, see Figure 4.29. Thus the environment affected the fatigue performance and it is felt that the α -case on the surface, due to the oxidation, assisted this surface cracking. The final failure was very likely caused by one of these cracks which grew into the sample, see Figure 4.29b. TiB-phase and α -phase cracking was observed in the fractured B-modified cast-and-extruded alloys similar to that observed for the B-modified as-cast alloys, see Figure 4.30.

Figure 4.28. (a)-(c) BSE SEMphotomicrographs taken from subsurface sections of post-fatigue as-cast Ti-64-xB samples. In (a), cracking within the α -phase laths was exhibited and periodic shear bands were evident in the β phase. Cracking within the TiB-phase was exhibited for the B-modified alloys in (b) and (c).



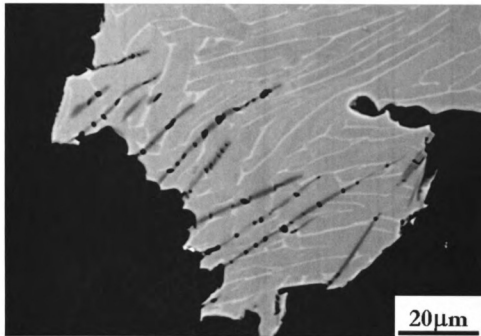
(a) Ti-64, $N_f=40,290$ cycles, $\sigma_{\max}=350$ MPa



(b) Ti-64-0.1B, $N_f=990,554$ cycles, $\sigma_{\max}=400$ MPa

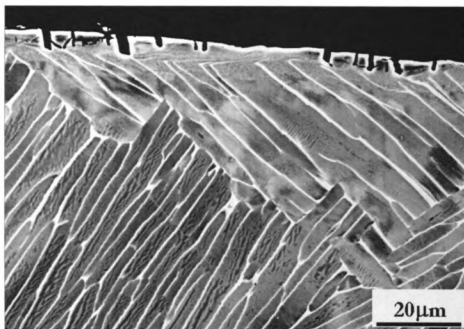
sections of pos-
hs was exhibit
e TiB-phase was

Figure 4.28 (continued)

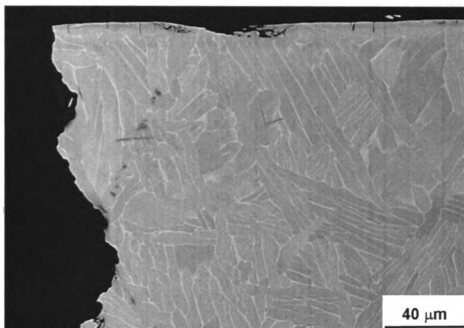


(c) Ti-64-1B, $N_f=32,718$ cycles, $\sigma_{\max}=350$ MPa

Figure 4.29. Post-fatigue BSE SEM photomicrographs showing secondary cracks in the α case on the surface of Ti-64-xB samples. Oxidation occurred during the fatigue tests.



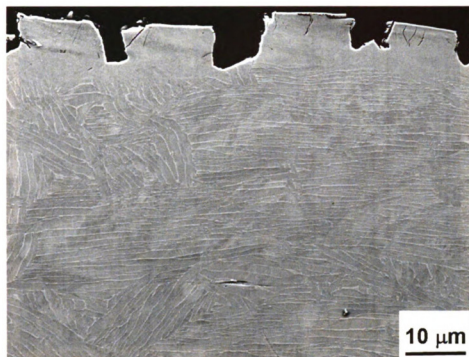
(a) As-cast Ti-64, $N_f=44,153$ cycles, $\sigma_{\max}=350$ MPa



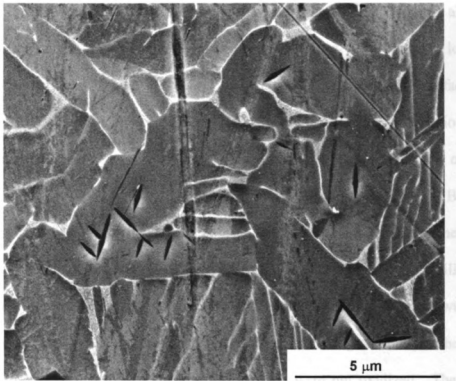
(b) As-cast Ti-64-0.1B, $N_f=990,554$ cycles, $\sigma_{\max}=400$ MPa

ary cracks in the
fatigue tests.

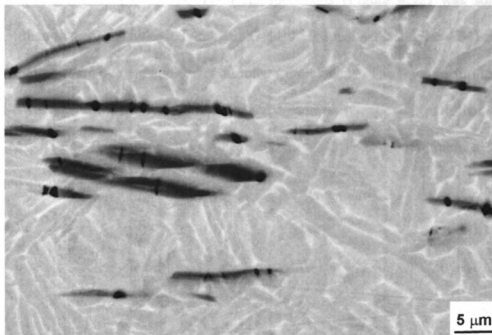
Figure 4.29 (continued)



(c) Cast-and-extruded Ti-64, $N_f=28,848$ cycles, $\sigma_{\max}=500$ MPa



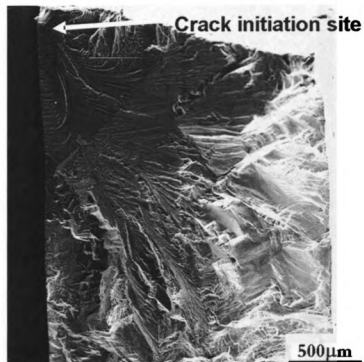
(a) Ti-64-0.1B, $N_f=20,351$ cycles, $\sigma_{\max}=550$ MPa



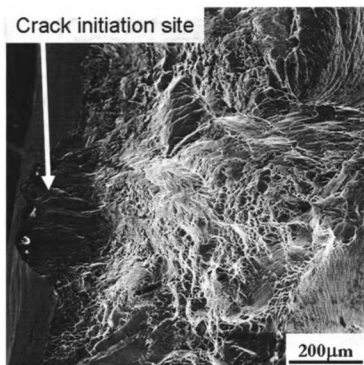
(b) Ti-64-1B, $N_f=79,230$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.30. Post-fatigue BSE SEM photomicrographs taken from subsurface sections of cast-and-extruded Ti-64-xB alloys. In (a), cracking within the α -phase laths was exhibited. In (b), cracking within the TiB-phase was exhibited.

SEM photomicrographs of fracture surfaces for the as-cast Ti-64-xB alloys are illustrated in Figures 4.31 to 4.34. Fracture initiation sites were typically located at surface locations, see Figures 4.31a and b. In some cases, multiple surface crack initiation sites were evident. Figure 4.31b illustrates the crack initiation, crack propagation, and overload regions for a Ti-64 sample. The overload regions contained ductile dimples, see for example Figures 4.32a and b for an as-cast Ti-64-1B sample. The crack growth regions of the sample consumed a large extent of the thickness of the samples, see Figure 4.33. Figure 4.33 illustrates the environmental susceptibility of the alloys as the colored region on the fracture surface indicated the crack growth region which oxidized during the experiment. The shiny metallic regions outside the colored region represent the overload regions/surfaces which were not oxidized. The fracture surface appearance of the as-cast Ti-64-1B specimens (Figure 4.32) was somewhat different than that for the as-cast Ti-64 and Ti-64-0.1B specimens (Figure 4.34) as larger faceted regions were evident and fewer dimples were evident. The fracture surface of as-cast Ti-64-0.1B (Figure 4.34) sample indicated a more tortuous crack path than that for the as-cast Ti-64-1B sample. In some cases, the as-cast Ti-64-1B fracture surfaces exhibited cleavage, indicating brittle fracture.

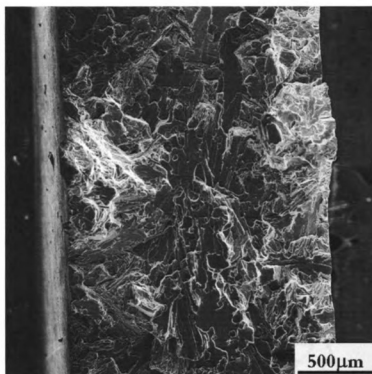


(a) As-cast Ti-64, $N_f=28,848$ cycles, $\sigma_{max}=350$ MPa

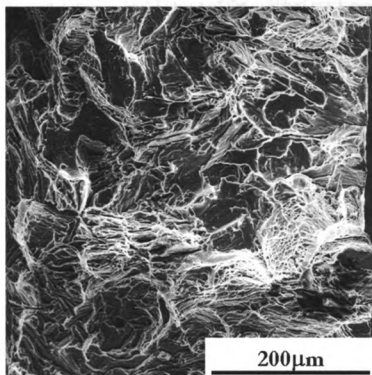


(b) As-cast Ti-64, $N_f=28,848$ cycles, $\sigma_{max}=350$ MPa

Figure 4.31. SE SEM photomicrographs of the fracture surface for an as-cast Ti-64 specimen. The crack initiation sites are indicated with arrows. This sample had two crack initiation sites. The overload region in (b) exhibited ductile dimples.



(a) As-cast Ti-64-1B, $N_f=32,718$ cycles, $\sigma_{\max}=350$ MPa



(b) As-cast Ti-64-1B, $N_f=32,718$ cycles, $\sigma_{\max}=350$ MPa

Figure 4.32. SE SEM photomicrographs of the fracture surface for an as-cast Ti-64-1B sample. The overload region in (b) exhibited ductile dimples.

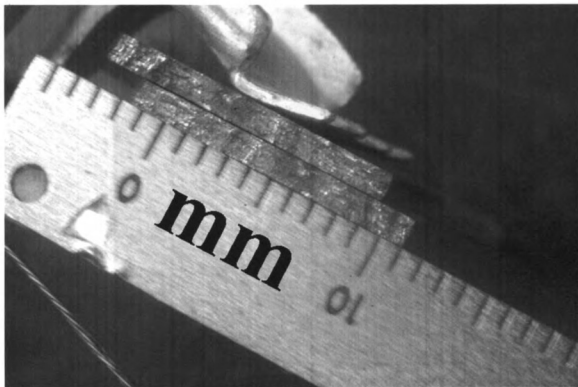
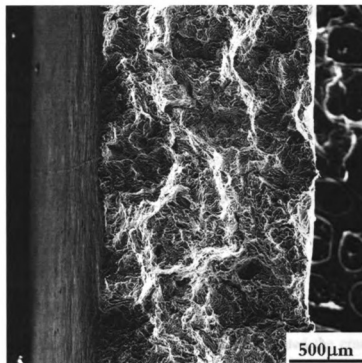
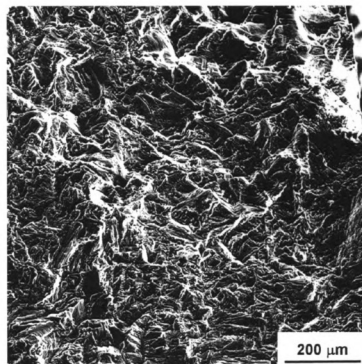


Figure 4.33. Low-magnification digital image of the oxidized crack growth region (dark colored) for an as-cast Ti-64-1B specimen which fractured after 25,555 cycles for a maximum applied stress of 400MPa. The ruler scale is in millimeters.



(a) As-cast Ti-64-0.1B, $N_f=990,554$ cycles, $\sigma_{\max}=400$ MPa



(b) As-cast Ti-64-0.1B, $N_f=990,554$ cycles, $\sigma_{\max}=400$ MPa

Figure 4.34. SE SEM photomicrographs of the fracture surface for an as-cast Ti-64-0.1B sample. (a) lower magnification and (b) higher magnification.

SEM photomicrographs of fracture surfaces for the cast-and-extruded Ti-64-xB alloys are illustrated in Figures 4.35 and 4.36. Fracture initiation sites were typically located at surface locations, see Figures 4.35a and b. Figure 4.36 clearly shows fatigue striations in the crack propagation region of a cast-and-extruded Ti-64 sample and a cast-and-extruded Ti-64-0.1B sample. In some cases, multiple surface crack initiation sites were evident. The overload regions in the cast-and-extruded Ti-64-xB alloys contained ductile dimples see for example Figures 4.37a, b and c, which indicates ductile failure. The crack growth regions of the sample consumed a large extent of the thickness of the samples, see Figure 4.38. Figure 4.38 illustrates the environmental susceptibility of the alloys as the colored region on the fracture surface indicated the crack growth region which oxidized during the experiment. The shiny metallic regions outside the colored region represent the overload regions/surfaces which were not oxidized.

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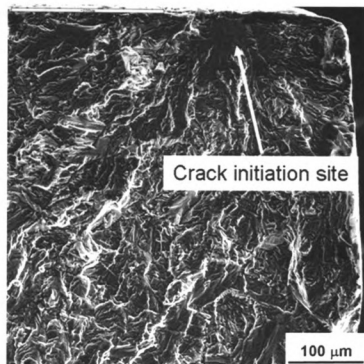
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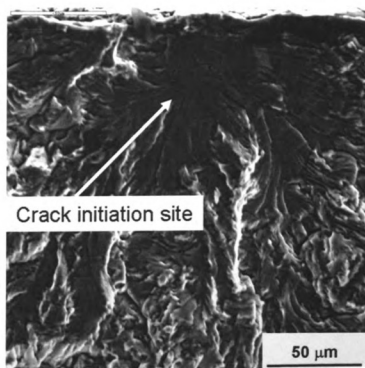
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TO AVOID FINES return on or before date due.
MAY BE RECALLED with earlier due date if requested.

DATE DUE	DATE DUE	DATE DUE

Figure 4.35. SE SEM photomicrographs of the fracture surface (a) (b) a cast-and-extruded Ti-64 sample. (c) (d) a cast-and-extruded Ti-64-1B sample. The crack initiation sites are indicated with arrows.

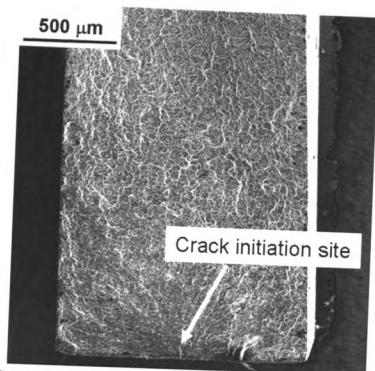


(a) Cast-and-extruded Ti-64, $N_f=28,848$ cycles, $\sigma_{\max}=500$ MPa

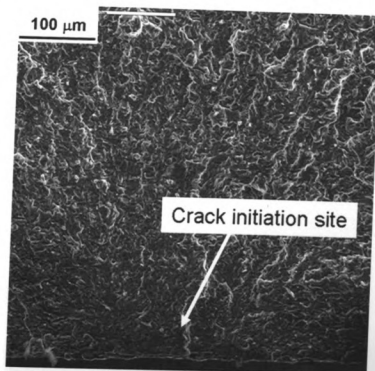


(b) Cast-and-extruded Ti-64, $N_f=28,848$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.35 (continued)

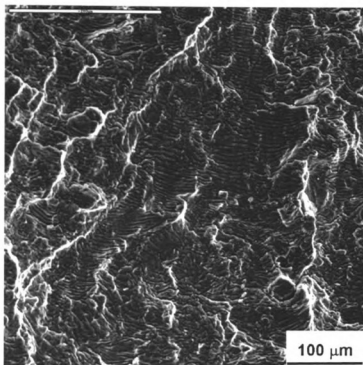


(c) Cast-and-extruded Ti-64-1B, $N_f=79,230$ cycles, $\sigma_{\max}=500$ MPa

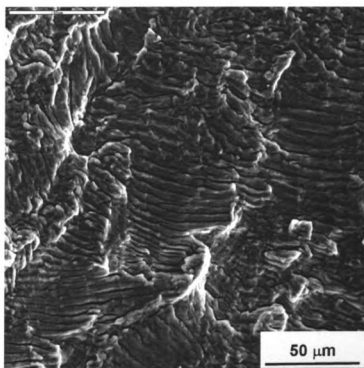


(d) Cast-and-extruded Ti-64-1B, $N_f=79,230$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.36. SE SEM photomicrographs of the fracture surface of (a) (b) a cast-and-extruded Ti-64 sample. (c) a cast-and-extruded Ti-64-0.1B sample. Fatigue striations are shown in the fatigue crack propagation region.

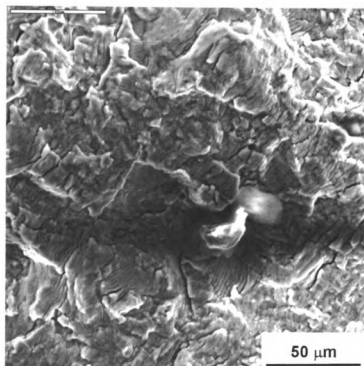


(a) Cast-and-extruded Ti-64, $N_f=28,848$ cycles, $\sigma_{\max}=500$ MPa



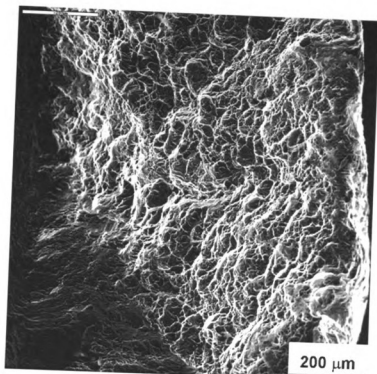
(b) Cast-and-extruded Ti-64, $N_f=28,848$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.36 (continued)

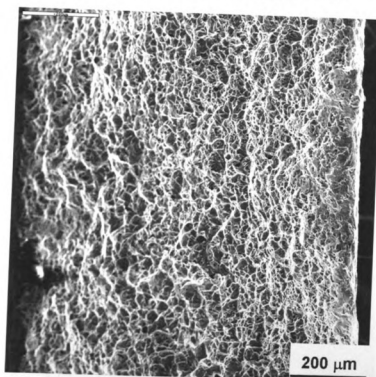


(c) Cast-and-extruded Ti-64-0.1B, $N_f=22,605$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.37. SE SEM photomicrographs of the overload region on fracture surface for cast-and-extruded Ti-64-xB alloys. The overload region exhibited ductile dimples.

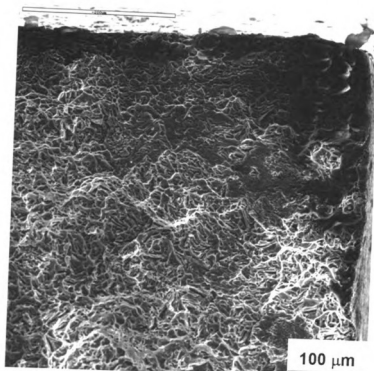


(a) Ti-64, $N_f=28,848$ cycles, $\sigma_{\max}=500$ MPa



(b) Ti-64-0.1B, $N_f=22,605$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.37 (continued)



(c) Ti-64-1B, $N_f=79,230$ cycles, $\sigma_{\max}=500$ MPa

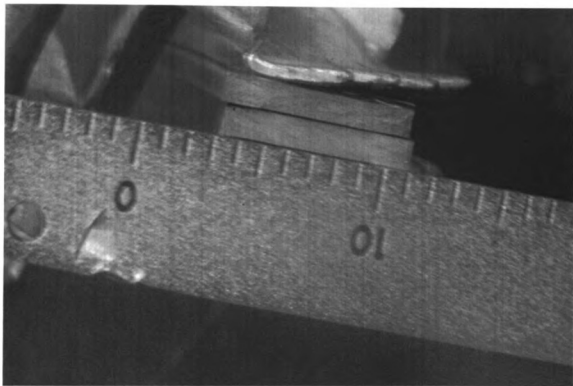
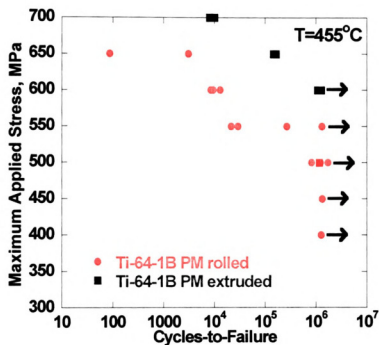


Figure 4.38. Low-magnification digital image of the oxidized crack growth region (dark colored) for a cast-and-extruded Ti-64-1B specimen which fractured after 42,127 cycles for a maximum applied stress of 500 MPa . The ruler scale is in millimeters.

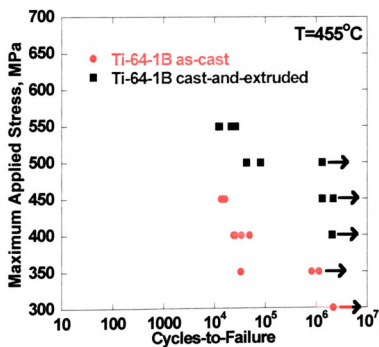
1.2 PM Processed Ti-64-1B Alloys

1.2.1 Fatigue Behavior (455°C)

The fatigue S-N curves obtained for the Ti-64-1B alloy with different processing conditions are shown in Figures 4.39a and b. For the PM processed alloys (Figure 4.39a), the extruded Ti-64-1B alloy exhibited significantly longer N_f values than the PM rolled Ti-64-1B alloy. Comparing with the N_f values of the as-cast and cast-and-extruded Ti-64-xB alloys, the fatigue N_f values of PM extruded Ti-64-1B were greater. The PM rolled Ti-64-1B alloy exhibited similar fatigue lives to those of the cast-and-extruded Ti-64-1B alloy. The N_f values of the as-cast Ti-64-1B were the lowest.



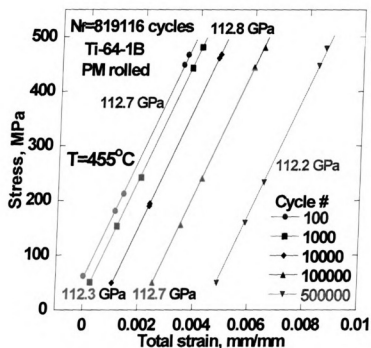
(a)



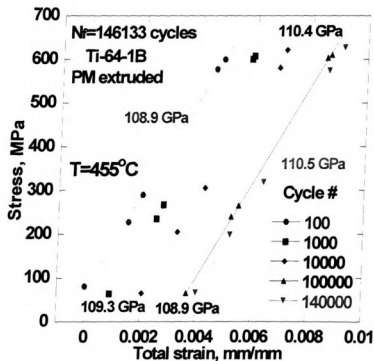
(b)

Figure 4.39. Maximum applied stress versus cycles to failure curves for the (a) PM rolled and PM extruded Ti-64-1B alloys, (b) as-cast and cast-and-extruded Ti-64-1B alloys. Runout specimens are marked with an arrow.

For the fatigue samples which fractured, stress versus strain plots during defined fatigue cycles are shown in Figure 4.40. No PM samples exhibited an E value reduction during the experiments, which suggested that little fatigue damage occurred. Selected fatigue runout samples were tensile tested at 455°C, see Table 4.14. Comparing these results to those in Table 4.10, it is evident that there was no strength loss due to fatigue. This confirms that little fatigue damage occurred to the samples that experienced runout. The shift of the hysteresis with increasing cycle number indicates creep occurred during the fatigue tests. This increased creep is reasonable based on the 455°C creep behavior of these alloys, as will be discussed later. No phase instability was observed on the post-fatigue gage sections for any of the samples tested even after more than 1.7 million cycles of fatigue exposure at 455°C. In such cases the measured volume percents of the TiB, α , and β phases were similar both before and after the experiments.



(a)



(b)

Figure 4.40. Stress versus strain plots at the marked cycle numbers for the studied alloys at 455°C. (a) PM rolled Ti-64-1B, and (b) PM extruded Ti-64-1B. The slope of each curve is indicated by the listed Young's modulus value.

Table 4.14. Average 455°C Tensile Properties of the Runout PM Processed Ti-64-1B Alloys

Alloy	Processing	E, GPa	YS, MPa	UTS, MPa	ϵ_f , %
Ti-64-1B	PM rolled	104	565	718	9.5
Ti-64-1B	PM extruded	115	716	836	8.7

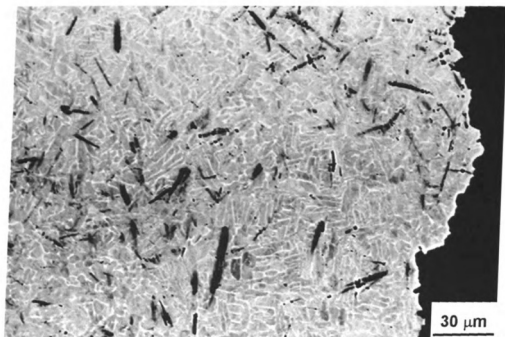
1.2.2 Fracture Analysis

For the PM processed Ti-64-1B alloys, TiB-phase cracking was evident within the deformed gage sections, see Figure 4.41.

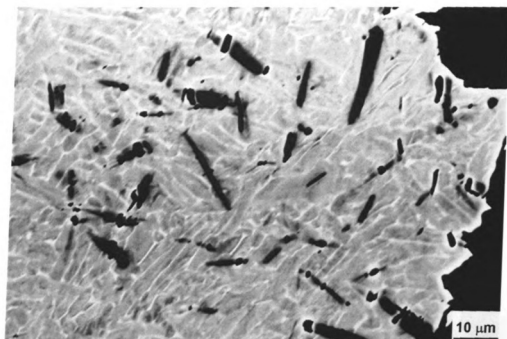
Similar to the as-cast and cast-and-extruded Ti-64-xB alloys, the fracture initiation sites of the PM processed Ti-64-1B alloys were typically located at surface locations. For one of the PM rolled samples, the crack initiated within two TiB whiskers close to the sample surface (Figure 4.42a). The direction of these two TiB whiskers was perpendicular to the loading direction. Cracks initiated from TiB whiskers perpendicular to the loading direction was also observed during the in-situ fatigue test for the as-cast Ti-6242S-0.4B alloy. Figure 4.42b illustrates the overload region of the PM rolled Ti-64-1B sample fracture surface which contained ductile dimples. Figure 4.43 is the fatigue fracture surface of a PM extruded Ti-64-1B sample. Most of the fracture surface exhibited ductile dimples and the crack initiation site was difficult to locate.



Figure 4.41. BSE SEM photomicrographs taken from subsurface sections close to the fatigue fracture surface of a (a) (b) Ti-64-1B PM rolled sample ($N_f=266,638$ cycles, $\sigma_{max}=550$ MPa). (c) (d) Ti-64-1B PM extruded sample ($N_f=162,628$ cycles, $\sigma_{max}=650$ MPa). TiB cracking is evident.

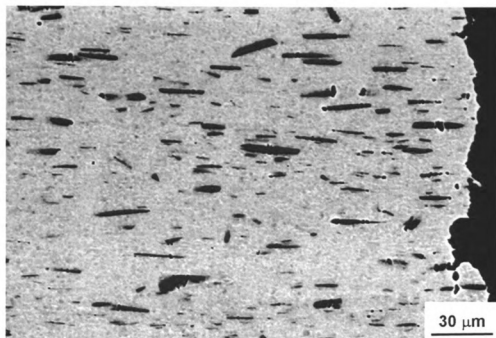


(a)

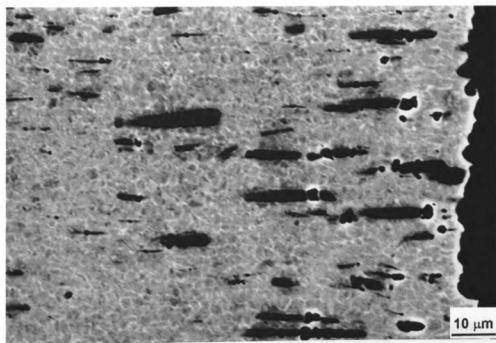


(b)

Figure 4.41 (continued)



(c)



(d)

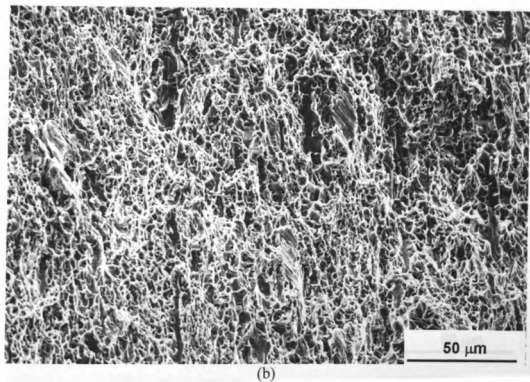
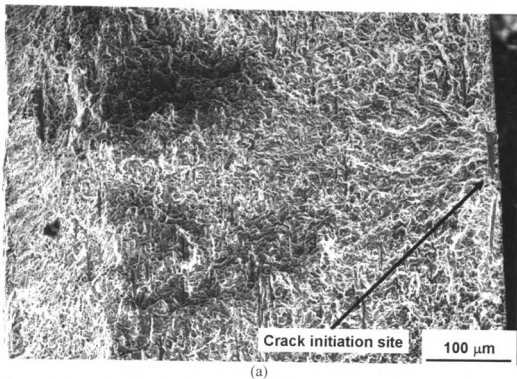


Figure 4.42. SE SEM photomicrographs of the fatigue fracture surface for a Ti-64-1B PM rolled specimen ($N_f=266,638$ cycles, $\sigma_{\max}=550$ MPa). The crack initiation sites are indicated with arrows in (a). The overload region in (b) exhibited ductile dimples.

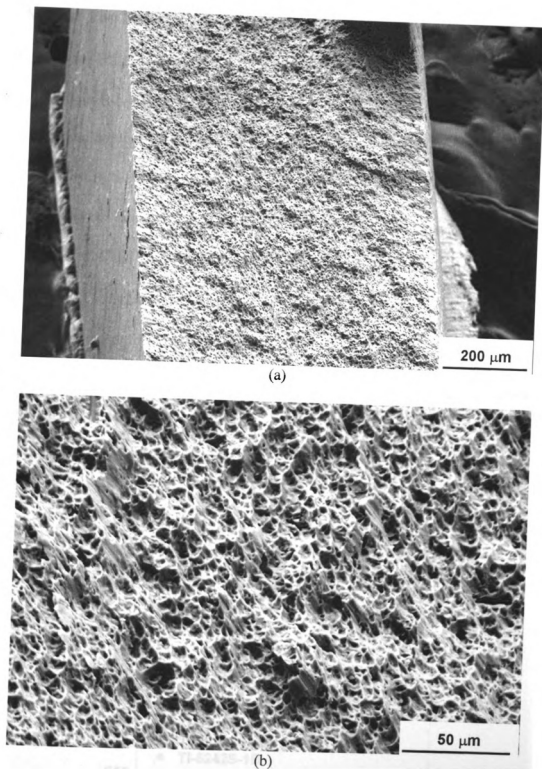


Figure 4.43. SE SEM photomicrographs of the fatigue fracture surface for a Ti-6Al-4V PM extruded specimen ($N_f=162,628$ cycles, $\sigma_{\max}=650$ MPa). (a) Low-magnification image, and (b) high-magnification image. Most of the fracture surface contained ductile dimples.

2. As-cast Ti-6242S-xB Alloy System

2.1 Fatigue Behavior (455°C)

Fatigue S-N curves for each Ti-6242S-xB alloy are illustrated in Figure 4.44. For the maximum applied stress levels of 350 MPa, 400 MPa, and 450 MPa, the Ti-6242S-0.1B and Ti-6242S-0.4B alloys exhibited longer average N_f values than Ti-6242S-1B and Ti-6242S alloys. Thus small B contents of less than 1 wt% were beneficial to the fatigue life. The Ti-6242S-1B alloy exhibited the lowest average N_f values at any given stress level. The Ti-6242S and Ti-6242S-1B alloys exhibited similar average N_f values compared to those for the Ti-64 and Ti-64-1B alloys (Figure 4.26a), respectively. However the Ti-6242S-0.1B alloy exhibited significantly longer average N_f values than the Ti-64-0.1B alloy.

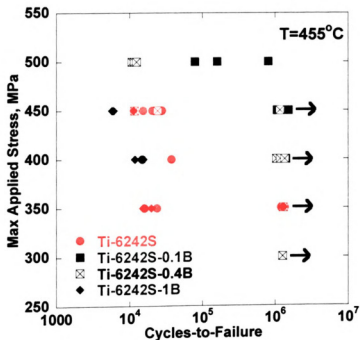


Figure 4.44. Maximum applied stress versus cycles-to-failure curves for the as-cast Ti-6242S-xB alloys where runout specimens are marked with an arrow.

Stress versus strain plots of selected Ti-6242S-xB alloy samples during defined fatigue cycles are illustrated in Figure 4.45. Typically there was not a sharp E value decrease with increasing cycle until the last few thousand cycles of the fatigue life. For all of the runout samples, an E value decrease during the experiment was not observed. No phase instability was observed on the post-fatigue gage sections for any of the samples tested even after more 1.4 million cycles of fatigue exposure at 455°C. In such cases the measured volume percents of the TiB, α , and β phases were similar both before and after the experiments.

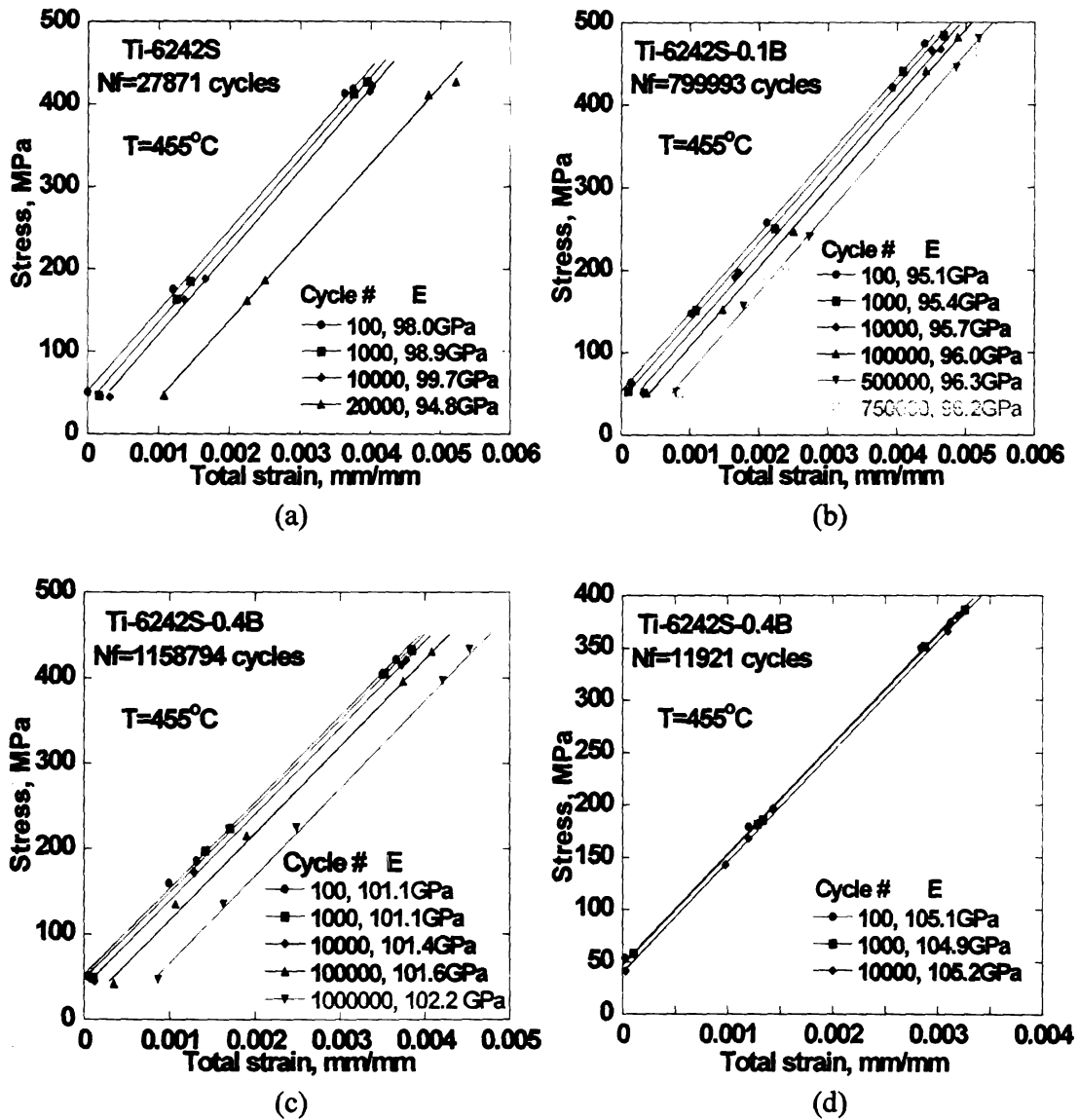


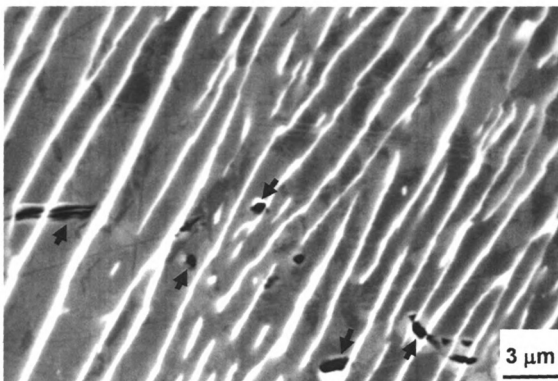
Figure 4.45. Stress versus strain plots at indicated cycles for as-cast Ti-6242S-xB alloys at 455°C.

2.2 Fracture Analysis

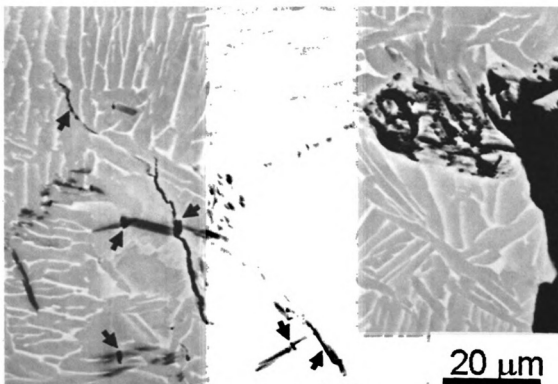
For the B-containing alloys, TiB-phase cracking was evident within the deformed gage sections, see Figures 4.46b, c and d. This behavior was similar to that for the Ti-64-xB system (Figures 4.28 and 4.41). Cracking within the TiB phase eventually led to cracking in the $\alpha+\beta$ phase matrix, see Figure 4.46b. The α -phase exhibited a significant amount of cracking, see Figure 4.46a, while cracks were only rarely observed within the fine β phase.

For each alloy, cracks were observed along the edges of the samples and grew perpendicular to the loading direction. Thus the environment affected the fatigue performance and it is felt that the α -case on the surface, due to the oxidation, assisted this surface cracking, see Figure 4.47. Similar to that for the Ti-64-xB alloy system, fracture initiation sites were typically located at surface locations, see Figure 4.48. In some cases, multiple surface crack initiation sites were evident. Figure 4.49 clearly shows fatigue striations in the crack propagation region of an as-cast Ti-6242S sample. For the Ti-6242S-1B alloy illustrated in Figure 4.48b, fatigue crack propagation was quite rapid, as will be discussed in the next section involving the fatigue crack growth behavior. Thus, once fatigue crack initiation was achieved, the Ti-6242-1B alloy's fatigue life was almost over. The fatigue crack propagation region within the Ti-6242S-0.1B and Ti-6242S-0.4B alloys was tortuous and ductile dimpling was evident in the overload regions, see Figures 4.50b and c.

Figure 4.46. Post-fatigue BSE SEM photomicrographs of as-cast Ti-6242S-xB alloys cracking within the α -phase laths was exhibited as indicated by the arrows in (a). Cracking within the TiB-phase was exhibited for the B-modified alloys in as indicated by the arrows (b), (c) and (d).

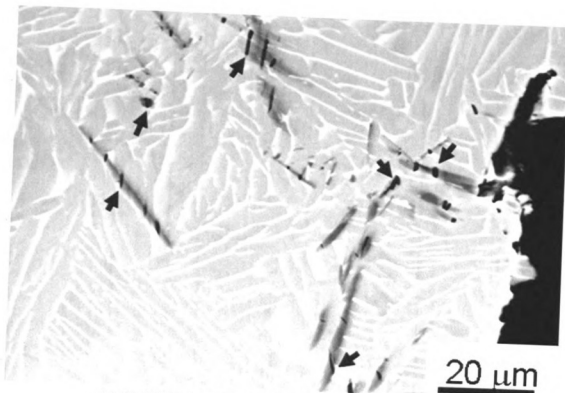


(a) Ti-6242S, $N_f=20,531$ cycles, $\sigma_{max}=450$ MPa

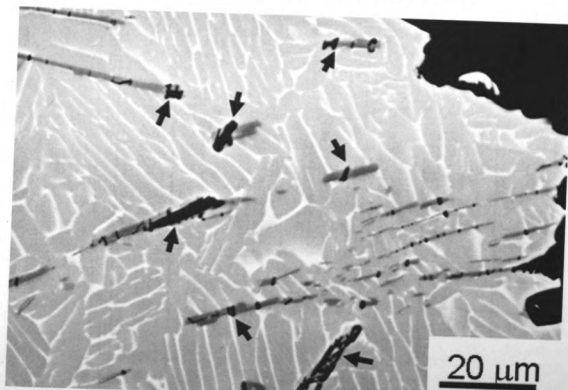


(b) Ti-6242S-0.1B, $N_f=78,404$ cycles, $\sigma_{max}=500$ MPa

Figure 4.46 (continued)

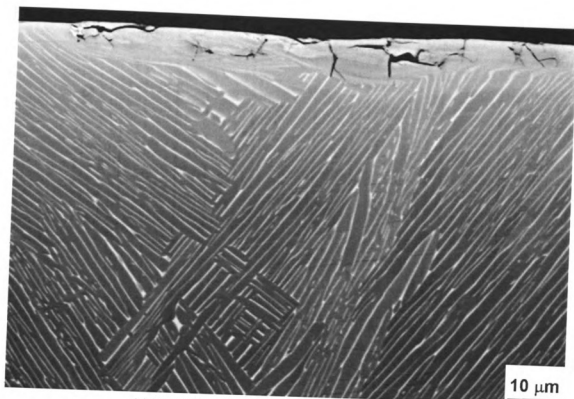


(c) Ti-6242S-0.4B, $N_f=1,158,794$ cycles, $\sigma_{\max}=450$ MPa

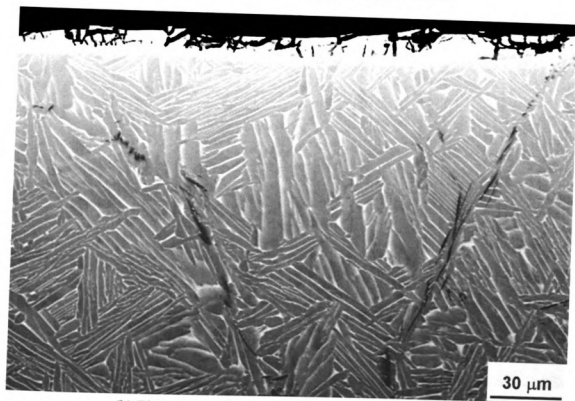


(d) Ti-6242S-1B, $N_f=14,894$ cycles, $\sigma_{\max}=400$ MPa

Figure 4.47. Post-fatigue BSE SEM photomicrographs of the edges for as-cast Ti-6242S-xB alloys. α -case formed during the fatigue test due to oxidation and secondary cracks were evident in the α -case.

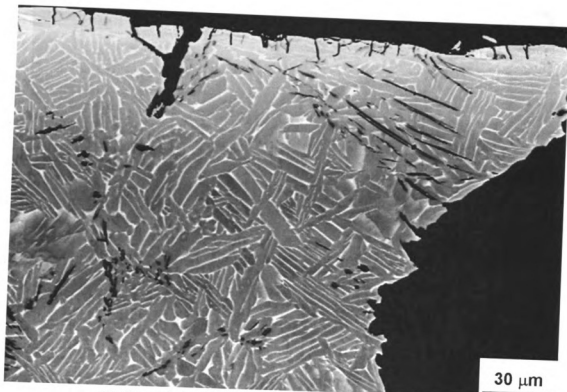


(a) Ti-6242S, $N_f=20,531$ cycles, $\sigma_{\max}=450$ MPa

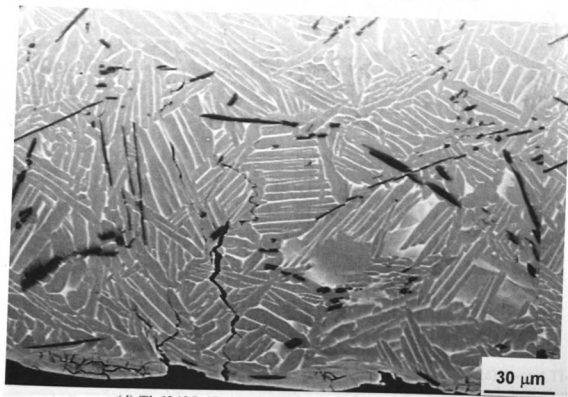


(b) Ti-6242S-0.1B, $N_f=78,404$ cycles, $\sigma_{\max}=500$ MPa

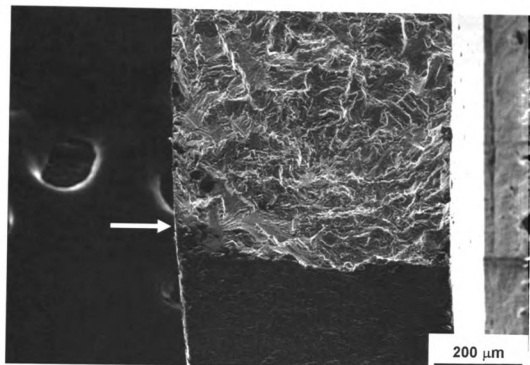
Figure 4.47 (continued)



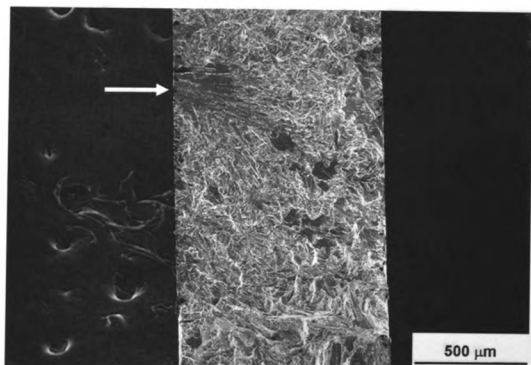
(c) Ti-6242S-0.4B, $N_f=1,158,794$ cycles, $\sigma_{\max}=450$ MPa



(d) Ti-6242S-1B, $N_f=14,894$ cycles, $\sigma_{\max}=400$ MPa

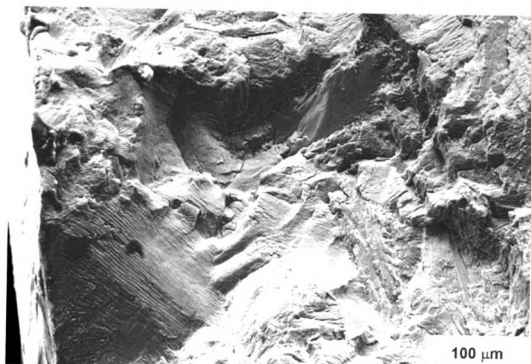


(a) Ti-6242S-0.1B, $N_f=78,404$ cycles, $\sigma_{\max}=500$ MPa

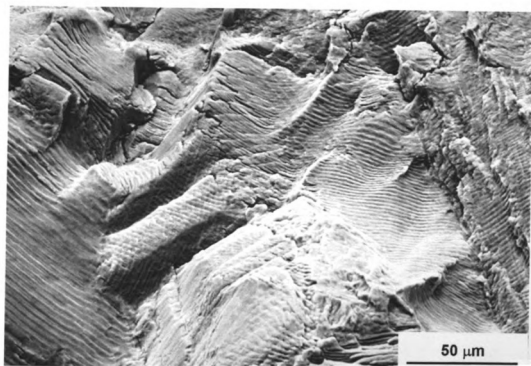


(b) Ti-6242S-1B, $N_f=19,894$ cycles, $\sigma_{\max}=350$ MPa

Figure 4.48. SE SEM photomicrograph of the fatigue fracture surface for (a) as-cast Ti-6242S-0.1B sample, and (b) as-cast Ti-6242S-1B sample. The fatigue crack initiation sites are indicated by the arrows.



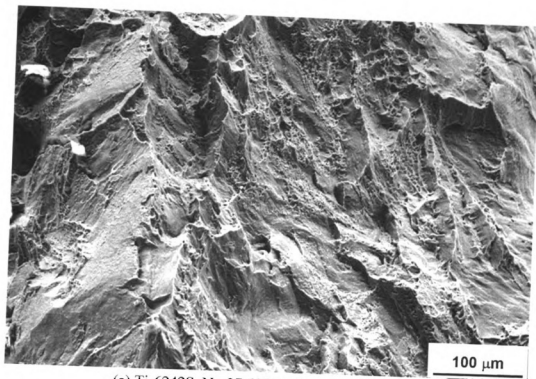
(a)



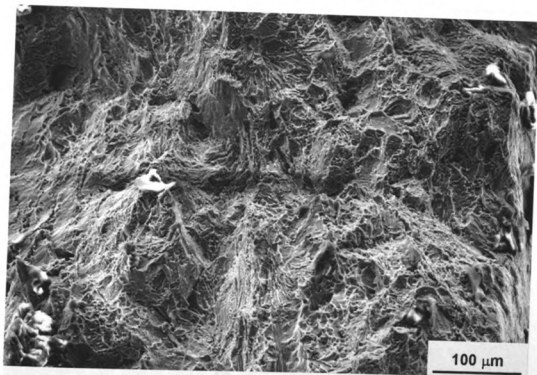
(b)

Figure 4.49. SE SEM photomicrographs of (a) (b) the fracture surface for an as-cast Ti-6242S sample which failed after 37,607 cycles at a maximum stress of 350 MPa. Fatigue striations are shown in the crack propagation region.

Figure 4.50. SE SEM photomicrographs of the fatigue fracture surface for as-cast Ti-6242S-xB alloys.

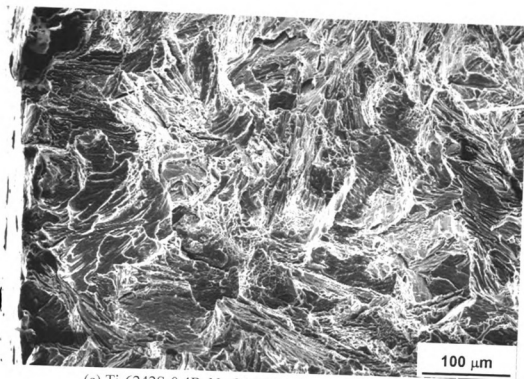


(a) Ti-6242S, $N_f=37,607$ cycles, $\sigma_{\max}=350$ MPa

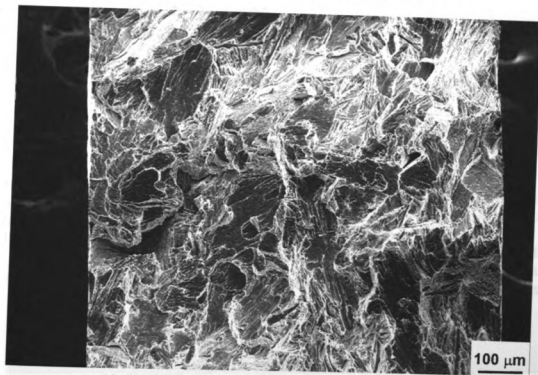


(b) Ti-6242S-0.1B, $N_f=78,404$ cycles, $\sigma_{\max}=500$ MPa

Figure 4.50 (continued)



(c) Ti-6242S-0.4B, $N_f=24,227$ cycles, $\sigma_{\max}=500$ MPa



(d) Ti-6242S-1B, $N_f=19,894$ cycles, $\sigma_{\max}=350$ MPa

Selected fatigue runout samples were tensile tested at 455°C and representative tensile stress versus strain plots are illustrated in Figure 4.51. Comparing these results to those in Figure 4.21, it is evident that little fatigue damage occurred during the experiments.

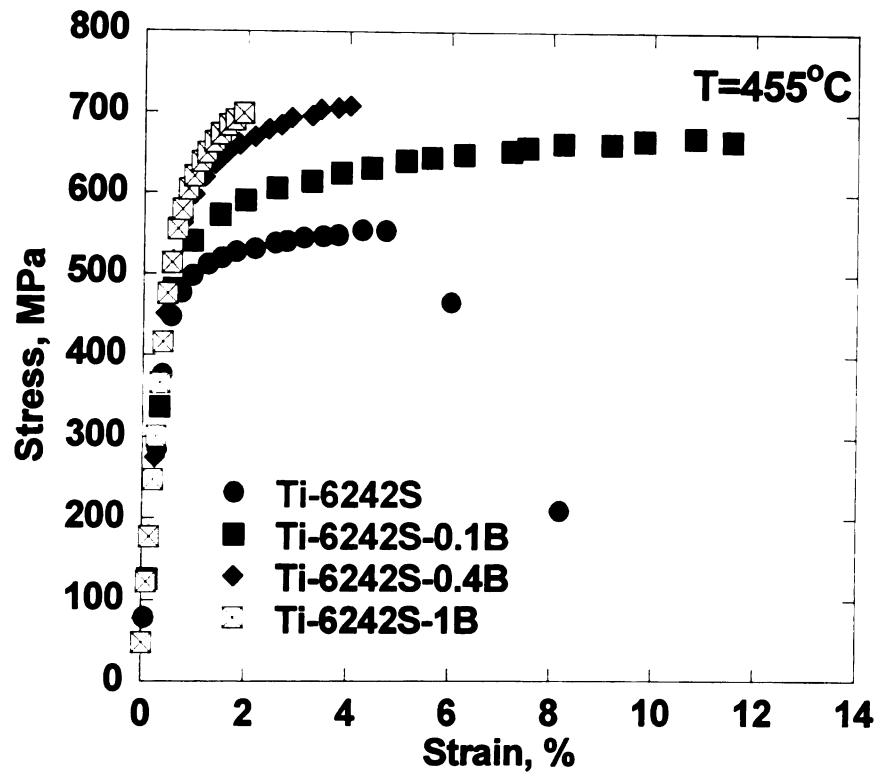
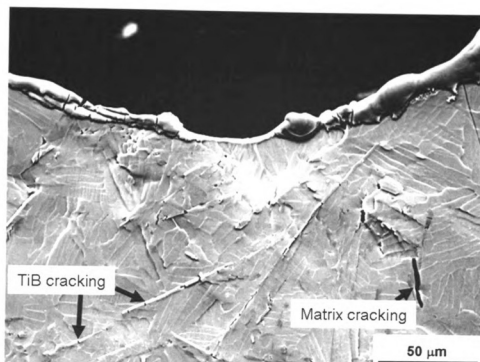


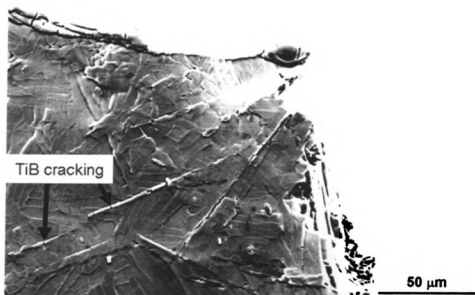
Figure 4.51. The 455°C tensile stress versus strain plot of as-cast Ti-6242S-xB alloys which have been subjected to fatigue testing at 455°C at the stress levels indicated in parenthesis and did not fail after more than one million cycles: Ti-6242S (400 MPa, 1.3 million cycles), Ti-6242S-0.1B (400 MPa, 1.4 million cycles), Ti-6242S-0.4B (300 MPa, 1.3 million cycles), Ti-6242S-1B (350 MPa, 1.3 million cycles). Comparing this to Figure 4.21 suggests that there was no fatigue damage occurred during the experiments.

2.3 Fatigue Crack Growth Behavior

The *in-situ* fatigue crack growth experiment performed on the Ti-6242S-1B alloy indicated that once the fatigue crack started from the notched region, it grew rapidly. In fact, the crack grew to final fracture of the specimen within the same cycle that it was observed, see Figure 4.52a and b. This suggests that this alloy exhibits poor fatigue crack growth resistance. The *in-situ* fatigue crack growth experiment performed on the Ti-6242S-0.4B alloy indicated that this alloy was more tolerant to fatigue cracking than the Ti-6242S-1B alloy. Figure 4.53a-h illustrates images of the surface deformation evolution of the notched Ti-6242S-0.4B specimen. In this case the cracks initially formed in the TiB whiskers and TiB/Ti-6242S interface ahead of the notch. Slip was then observed in the α -phase surrounding the cracked TiB phase followed by α -phase cracking. The surface slip behavior noted in the Ti-6242S-0.4B alloy was similar to that observed in previous fatigue crack growth studies of the Ti-6242S alloy [Sansoz (2003)].



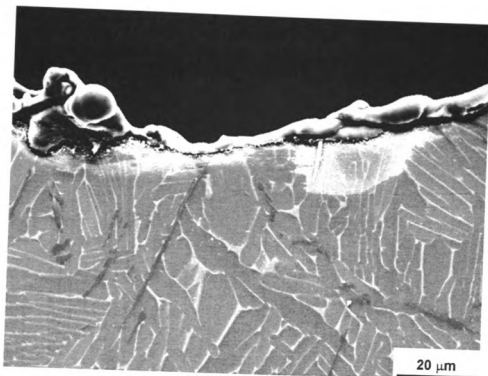
(a)



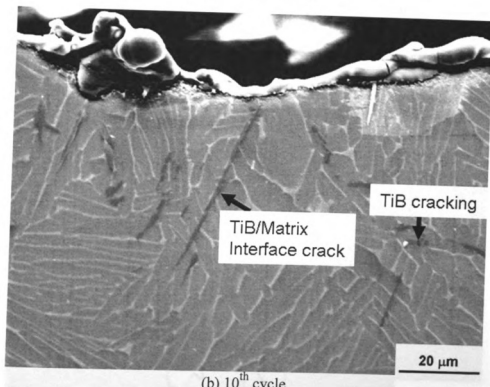
(b)

Figure 4.52. SE SEM photomicrographs for a notched Ti-6242S-1B sample fatigue tested at a maximum applied stress of 350 MPa. (a) 203rd cycle, and (b) post failure. The loading axis was horizontal in the images.

Figure 4.53. (a)-(h) SE SEM photomicrographs for a notched Ti-6242S-0.4B sample fatigue tested at a maximum stress of 400 MPa. The loading axis was horizontal in the images. Cracking ahead of the notch tip was evident and as the TiB/Matrix interface crack joined the notch the sample broke before reaching the 46th cycle.

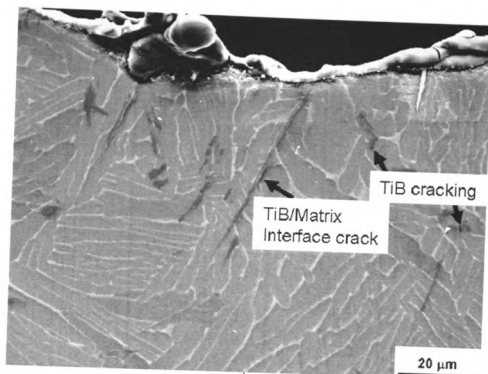


(a) Before loading

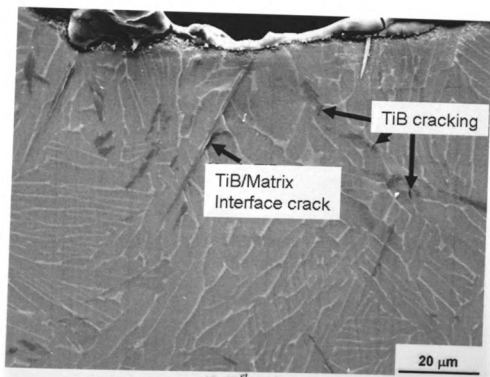


(b) 10th cycle

Figure 4.53 (continued)

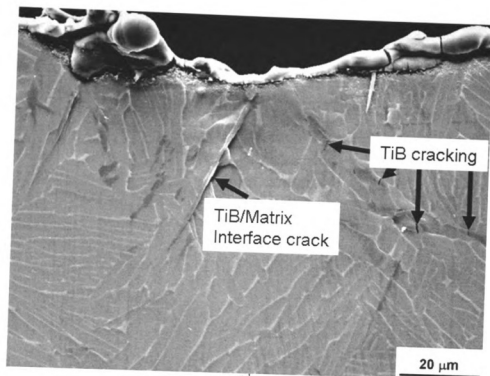


(c) 20th cycle

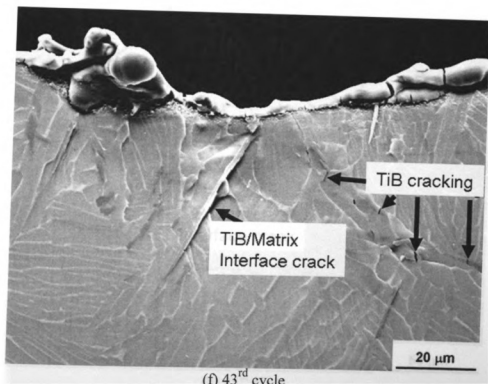


(d) 41st cycle

Figure 4.53 (continued)

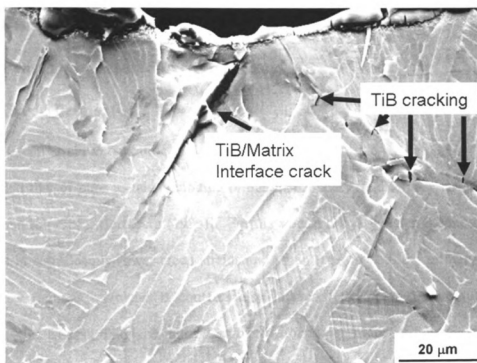


(e) 42nd cycle

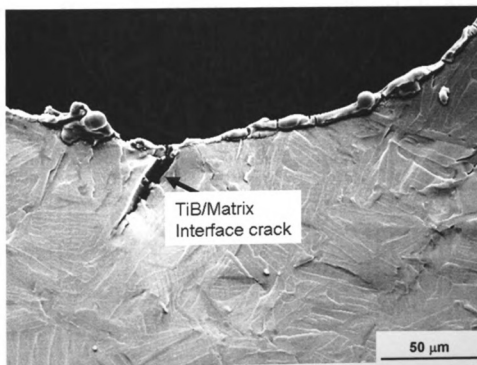


(f) 43rd cycle

Figure 4.53 (continued)



(g) 44th cycle



(h) 45th cycle

D. Creep Behavior Results

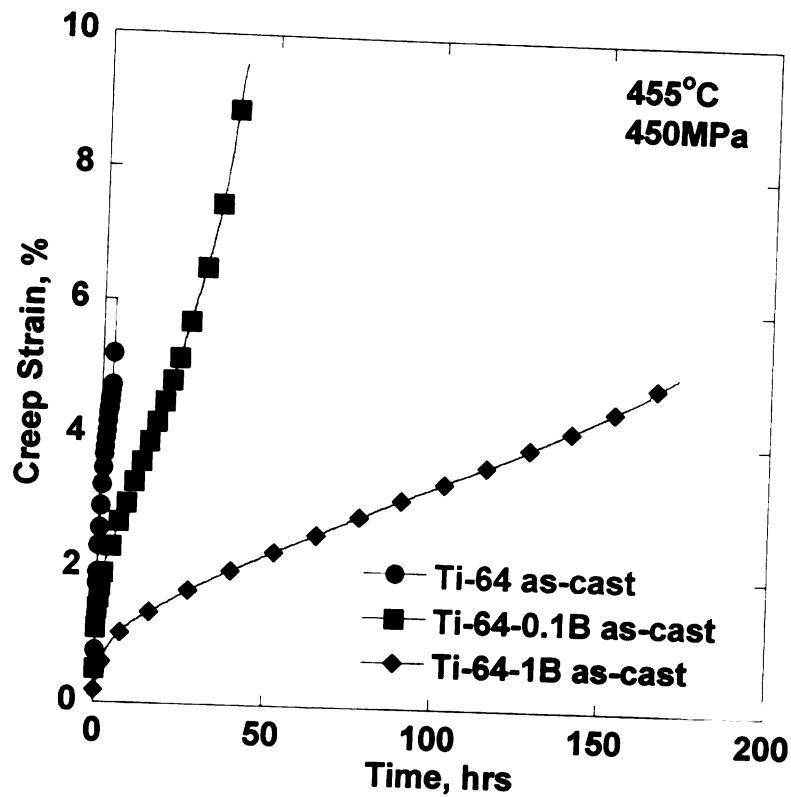
1. Ti-64-xB Alloy System

1.1 As-cast and Cast-and-extruded Alloys

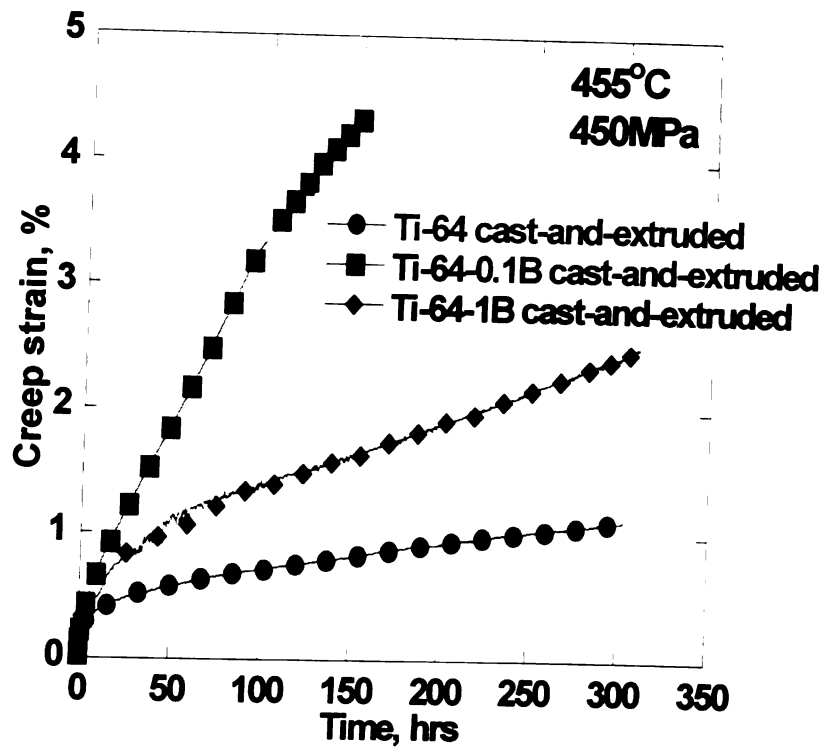
1.1.1 Creep Properties

Creep strain versus time curves are shown in Figure 4.54. The creep behavior resembled that of pure metals exhibiting primary, secondary, and tertiary stages [Evans and Wilshire (1985), Hertzberg (1996)]. For the as-cast alloys, greater B concentrations resulted in significantly lower creep strains and strain rates in both the primary and the secondary regimes. Greater B contents also increased the creep-rupture times. In general, the as-cast Ti-64-1B alloy exhibited around an order of magnitude lower minimum strain rate than the as-cast Ti-64 alloy for a given applied stress and temperature. The creep stress exponents (n), which were calculated using the minimum creep rate versus stress plots shown in Figure 4.55a, were greater than 8. The apparent activation energy (Q_{app}) values ranged between 275-395 kJ/mol at $\sigma=450$ MPa (see Figure 4.56a).

Figure 4.54. Creep strain versus time curves of the studied alloys at various temperatures and applied stress levels. at $T=455^{\circ}\text{C}$, $\sigma=450$ MPa: (a) as-cast (b) cast-and-extruded alloys; at $T=455^{\circ}\text{C}$, $\sigma=400$ MPa: (c) as-cast (d) cast-and-extruded alloys; at $T=400^{\circ}\text{C}$, $\sigma=450$ MPa: (e) as-cast (f) cast-and-extruded alloys.



(a)



(b)

Figure 4.54 (continued)

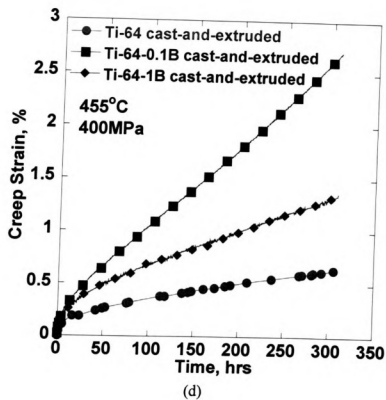
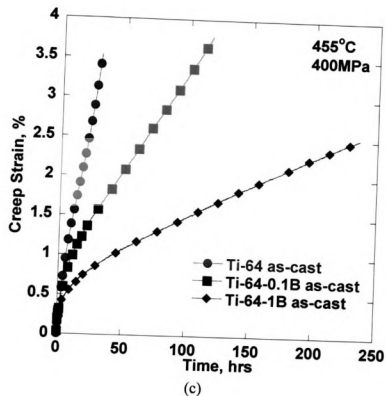
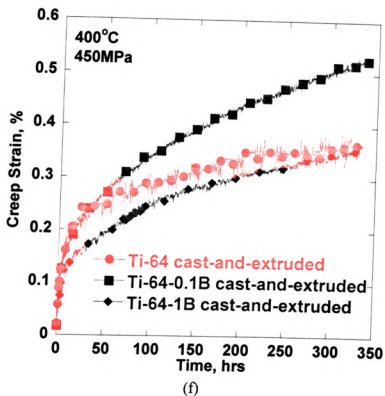
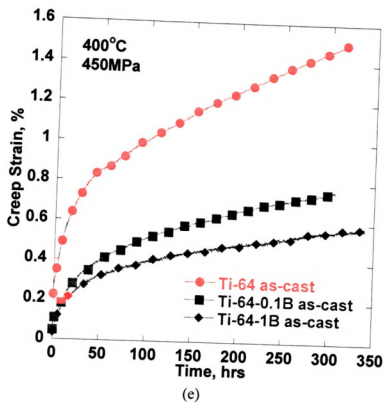
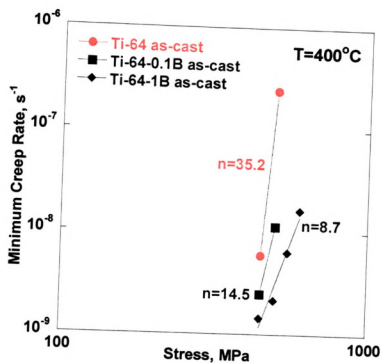


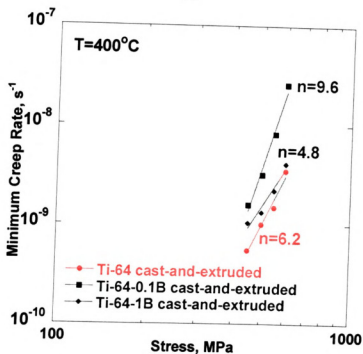
Figure 4.54 (continued)



For the cast-and-extruded alloys, the lowest secondary creep strain rates were exhibited by the Ti-64 alloy, while the Ti-64-1B alloy exhibited lower primary creep strains and secondary creep strain rates than the Ti-64-0.1B alloy (see Figure 4.54b, d, and f). The Ti-64-0.1B alloy always entered tertiary creep prior to the other alloys for a given applied stress and temperature. The n values were greater than or equal to 4.8 at 400°C, (see Figure 4.55b) and the Q_{app} values ranged between 287-301 kJ/mol at $\sigma=600$ MPa for temperatures between 400-455°C (see Figure 4.56b). For both microstructures of the as-cast and cast-and-extruded alloys, no phase instability was observed on the post-creep gage sections even after more than 865 hours of creep exposure at 400°C. In such cases the measured volume percents of the TiB, α , and β phases were similar both before and after the experiments.



(a)



(b)

Figure 4.55. Minimum creep rate ($\dot{\epsilon}_{\min}$) versus stress (σ) plot at $T=400^{\circ}\text{C}$ for Ti-64, Ti-64-0.1B, and Ti-64-1B (a) as-cast and (b) cast-and-extruded alloys illustrating the variation of minimum creep rate with stress. The creep stress exponent (n) values determined from these plots are shown.

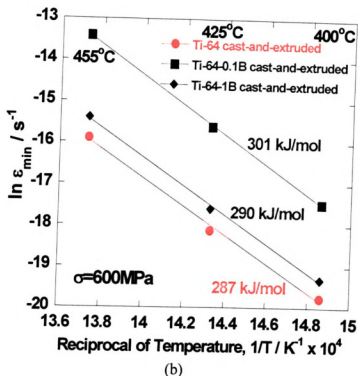
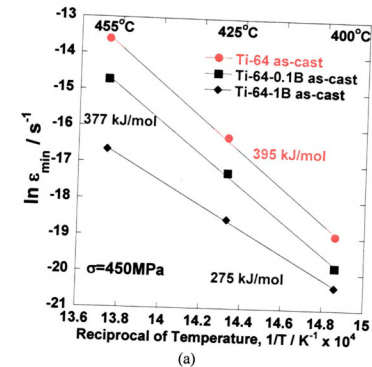


Figure 4.56. $\ln \dot{\epsilon}_{\min}$ versus reciprocal of temperature for the (a) as-cast alloys at $\sigma=450$ MPa and for the (b) cast-and-extruded alloys at $\sigma=600$ MPa illustrating the variation of minimum creep rate with temperature. Q_{app} values determined from these plots are shown.

Deformation observations indicated that extensive TiB whisker cracking was evident, and the extent of the TiB whisker cracking increased with increased creep strain. BSE SEM images taken from polished gage sections of the deformed creep specimens (see Figures 4.57 and 4.58) indicated the extent of the TiB whisker cracking within the bulk of the material. Cracking was also observed within the α phase and at the interfaces between the α and β phases (see Figure 4.59). In addition environmentally-assisted cracking was evident at the edges of the samples, where an α -case layer had developed due to oxidation at the surface (see Figure 4.60a and b). Such cracking occurred preferentially along the α -case layer at the surface, which was formed due to oxidation.

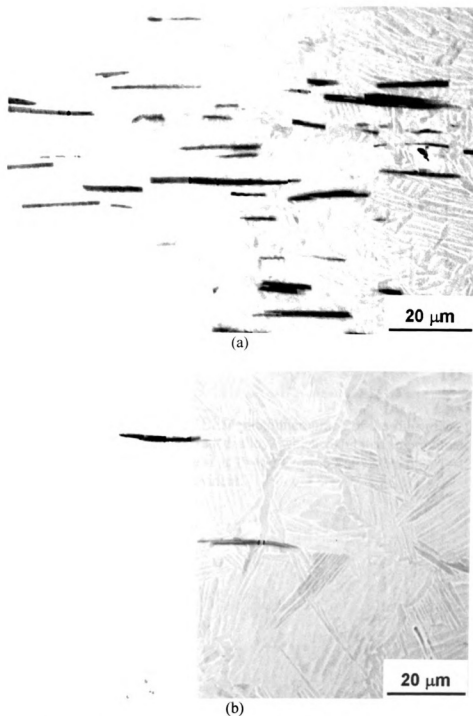
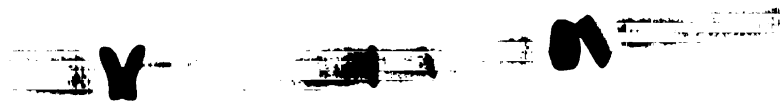


Figure 4.57. BSE SEM photomicrographs of the subsurface thickness sections of (a) a Ti-64-1B cast-and-extruded alloy, which exhibited a creep strain of 1.5% after undergoing a creep test at $T=455^{\circ}\text{C}/\sigma=400$ MPa, and (b) a Ti-64-0.1B cast-and-extruded alloy, which exhibited a creep strain of 2.8% after undergoing a creep test at $T=455^{\circ}\text{C}/\sigma=400$ MPa. The horizontal axis was the loading direction. The samples exhibited TiB whisker cracking.



10 μ m

Figure 4.58. High-magnification BSE SEM photomicrograph of the subsurface thickness section of a Ti-64-0.1B cast-and-extruded alloy, which exhibited approximately 5.2% creep strain after undergoing a creep test at $T=400^{\circ}\text{C}/\sigma=450\text{-}600\text{ MPa}$ then $T=425^{\circ}\text{C}$ and $T=455^{\circ}\text{C}$. TiB whisker cracking was evident.

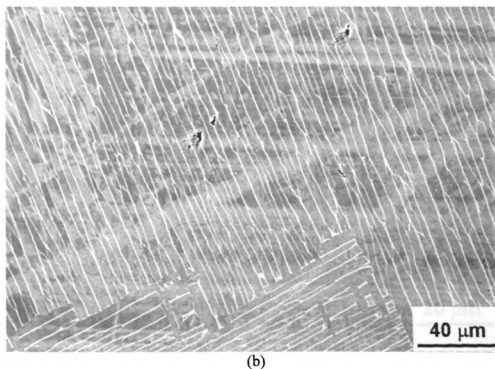
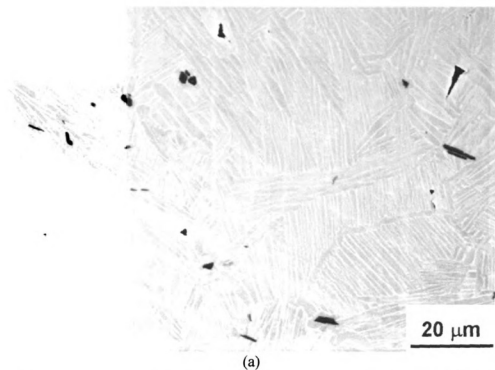
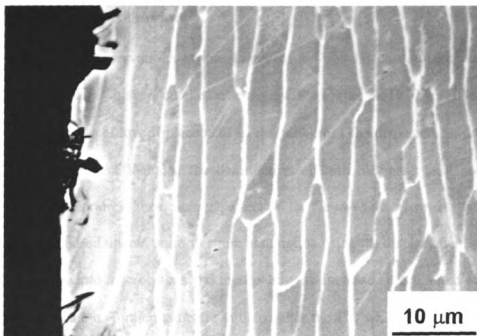
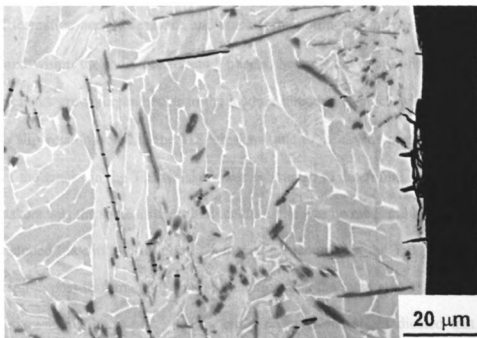


Figure 4.59. BSE SEM photomicrographs of the subsurface thickness section indicating cracking in the $\alpha+\beta$ microstructure for creep samples tested at $T=455^{\circ}\text{C}$ / $\sigma=400$ MPa (a) a Ti-64-0.1B cast-and-extruded alloy, which exhibited approximately 3.8% creep strain and (b) a Ti-64 as-cast alloy, which exhibited approximately 3.5% creep strain. The cracks are identified by their dark black color.



(a)



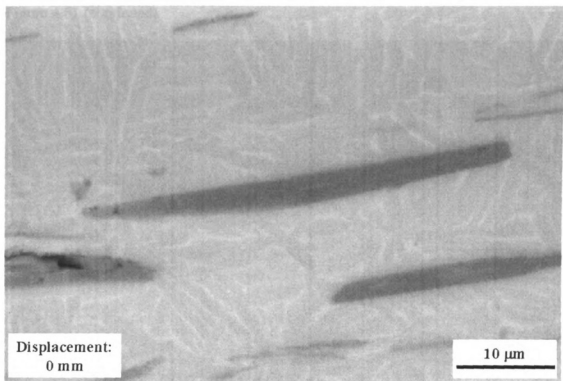
(b)

Figure 4.60. BSE SEM photomicrographs indicating environmentally-affected secondary cracks at the edges of the samples for (a) a Ti-64 as-cast alloy, which exhibited approximately 6.0% creep strain after a creep test at $T=455^{\circ}\text{C}$ / $\sigma=400$ MPa, and for (b) a Ti-64-1B as-cast alloy, which exhibited approximately 5.8% creep strain after a creep test at $T=400^{\circ}\text{C}$ / $\sigma=450$ -600 MPa then $T=425^{\circ}\text{C}$ and $T=455^{\circ}\text{C}$.

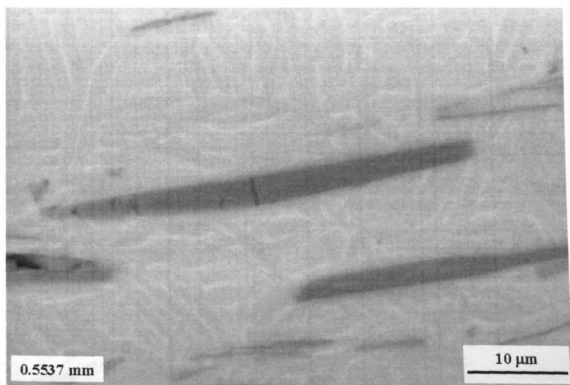
1.1.2 *In-situ* Creep Behavior

In-situ creep observations of the surface of a Ti-64-1B cast-and-extruded alloy indicated that the TiB whiskers cracked extensively (see Figure 4.61). The TiB whiskers exhibited microcracks at low displacement levels (500 μm) (The displacement during the *in-situ* creep experiment included the deformation of both the gauge and non-gauge section. Thus, the strain in the heated gage section for the *in-situ* experiments was likely greater than that based upon a uniform strain assumption using the displacement.). These microcracks grew into macrocracks and multiplied with increased creep strain and time. Localized slip in the $\alpha+\beta$ microstructure initiated after the TiB whisker cracking became extensive. Deformation continued via large plasticity in the $\alpha+\beta$ microstructure followed by void formation and coalescence and crack formation at the interface of the α and β phases and within the individual α and β phases, before final ductile fracture. The TiB whisker cracks grew perpendicular to the loading direction to crack widths as large as 3 μm . No slip was observed in the TiB whiskers, and to assess the possibility of slip, the length of the cracks in the TiB whiskers was subtracted to determine if the length of the TiB whiskers was the same as the initial length; there was no measurable change. Thus it was concluded that little-to-no plastic deformation occurred in the TiB phase. A lower-magnification post-test image of an area encompassing the same area exhibited in Figure 4.61 is shown in Figure 4.62. *In-situ* creep observations also indicated that the TiB whiskers cracked extensively for the Ti-64-0.1B cast-and-extruded alloy (see Figure 4.63) and the deformation evolution events were similar to that for the Ti-64-1B alloy.

Figure 4.61. BSE SEM photomicrographs for a Ti-64-1B cast-and-extruded sample *in-situ* creep tested at $T=455^{\circ}\text{C}/\sigma=450\text{ MPa}$. The creep displacements are indicated in the lower left hand side of the images (a)-(i). The loading axis was horizontal in the images.

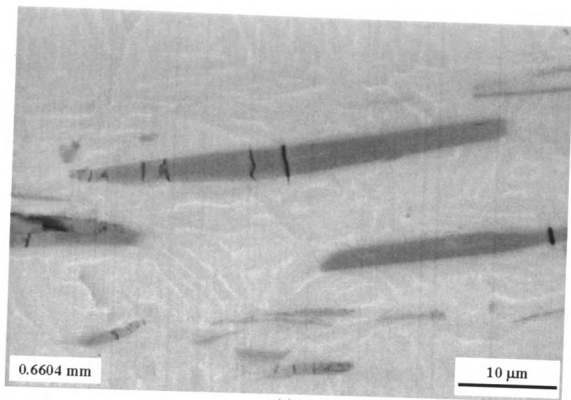


(a)

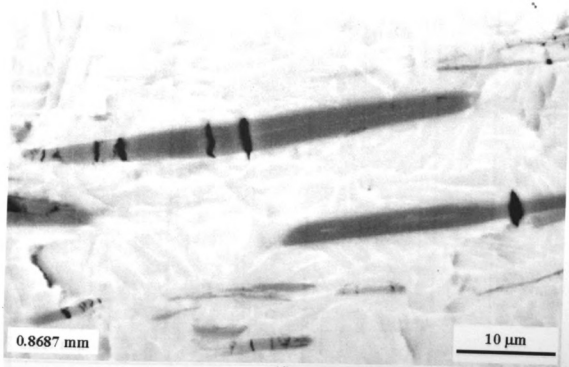


(b)

Figure 4.61 (continued)



(c)



(d)

Figure 4.61 (continued)

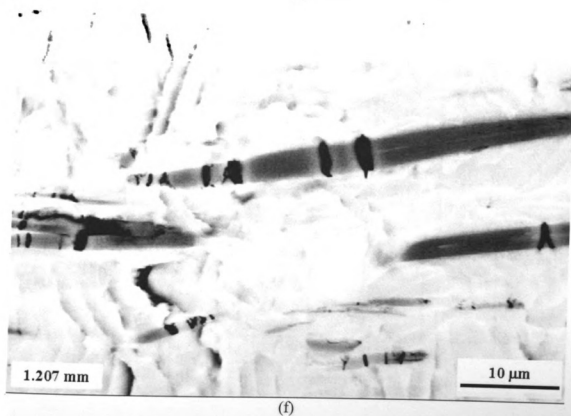
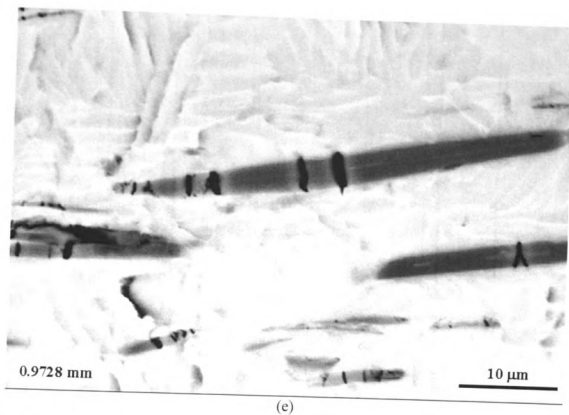
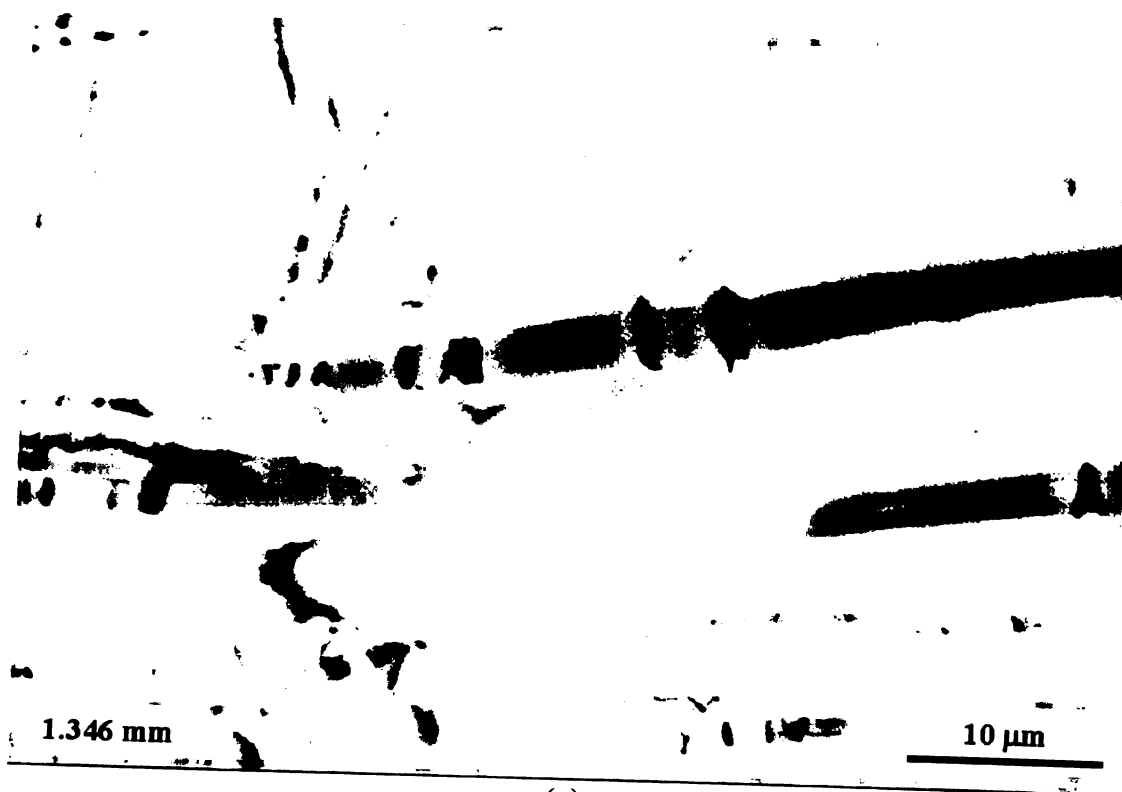


Figure 4.61 (continued)

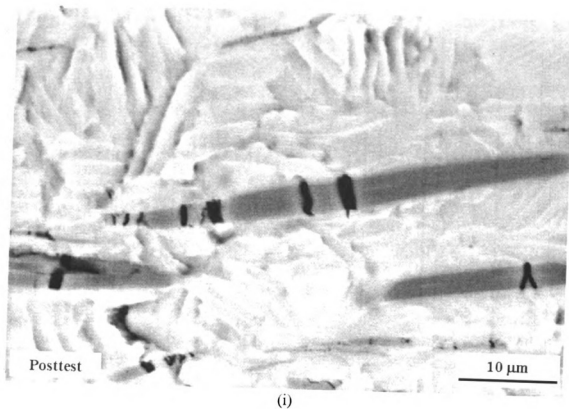


(g)



(h)

Figure 4.61 (continued)



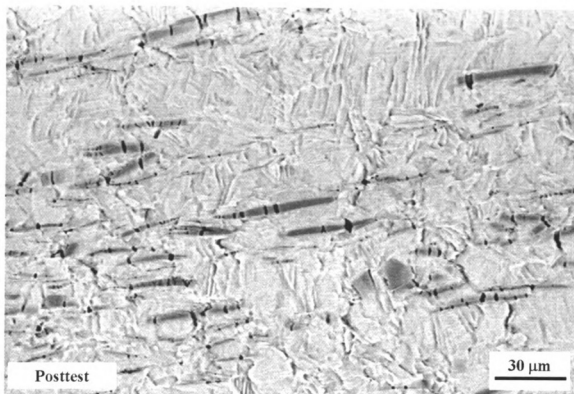


Figure 4.62. Lower-magnification BSE SEM photomicrograph of the Ti-64-1B cast-and-extruded sample depicted in Figure 4.61 after the test was completed. The loading axis was horizontal in the image.

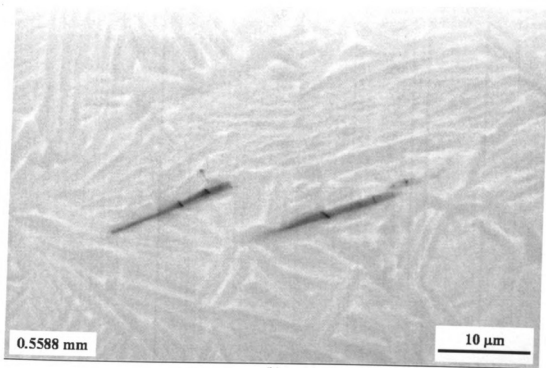
Figure 4.63. BSE SEM photomicrograph for a Ti-64-1B cast-and-extruded sample *in-situ* creep tested at $T=455^{\circ}\text{C}$, $\sigma=450\text{ MPa}$. The creep displacements are indicated in the lower left hand side of the images (a)-(f). The loading axis was horizontal in the images.



Displacement:
0.0 mm

10 μ m

(a)

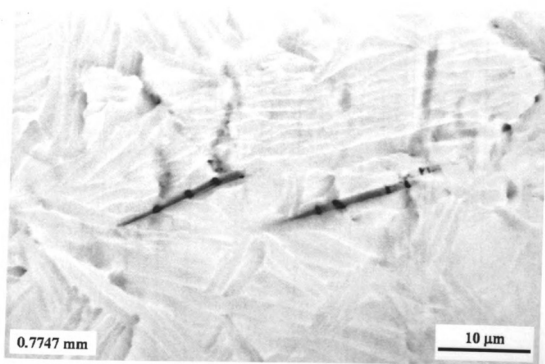


(b)

Figure 4.63 (continued)

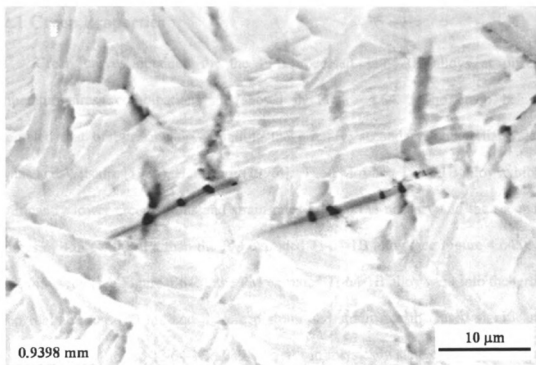


(c)

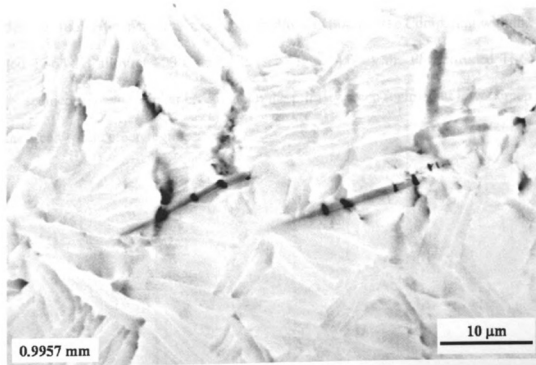


(d)

Figure 4.63 (continued)



(e)



(f)

1.2 PM Processed Ti-64-1B Alloys

1.2.1 Creep Properties

Creep curves obtained for the PM processed Ti-64-1B alloys at different temperature-stress combinations are shown in Figure 4.64. The creep behavior resembled that of pure metals exhibiting the primary, secondary, and tertiary stages [Evans and Wilshire (1985), Hertzberg (1996)]. The PM rolled Ti-64-1B alloy exhibited significantly lower creep strains and strain rates in both the primary and the secondary regimes at 455°C/400MPa than the PM extruded Ti-64-1B alloy (see Figure 4.64a). At 455°C/450MPa (see Figure 4.64b), the PM extruded Ti-64-1B alloy went into the tertiary creep regime after 100 hrs and the creep strain and strain rate increased significantly. However, the PM rolled Ti-64-1B alloy was still in the steady state creep regime with a lower creep rate. The PM rolled Ti-64-1B alloy did not go to the tertiary creep regime under the studied temperature/stress combinations in 300 hours. Comparing with the PM rolled Ti-64-1B alloy at 400°C/450MPa (see Figure 4.64c), the PM extruded Ti-64-1B alloy seemed to have a higher creep rate in the primary creep regime but lower creep rate in the steady-state creep regime.

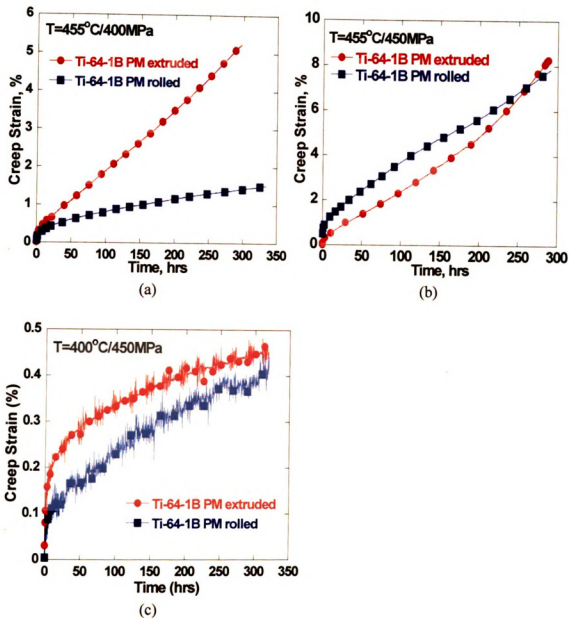


Figure 4.64. Creep strain versus time curves for the PM processed Ti-64-1B samples at (a) $T=455^{\circ}\text{C}$, $\sigma=400\text{ MPa}$. (b) $T=455^{\circ}\text{C}$, $\sigma=450\text{ MPa}$, and (c) $T=400^{\circ}\text{C}$, $\sigma=450\text{ MPa}$.

The creep stress exponent (n) calculated using the minimum creep rate versus stress plots shown in Figure 4.65, was 6.6 at 400°C and 10.7 at 455°C for the PM rolled Ti-64-1B alloy. The n value of PM extruded Ti-64-1B alloy was 2.5 at 455°C (based on two data points). The apparent activation energy values (Q_{app}) at 450 MPa was 257 kJ/mol for the PM rolled Ti-64-1B alloy and 293 kJ/mol for the PM extruded Ti-64-1B alloy (see Figure 4.66a). The Q_{app} for the PM rolled Ti-64-1B alloy at 550MPa was 261 kJ/mol (see Figure 4.66b). For both the PM processed Ti-64-1B alloys' microstructures, no phase instability was observed on the post-creep gage sections. In such cases the measured volume fractions of the TiB, α , and β phases were similar both before and after the experiments.

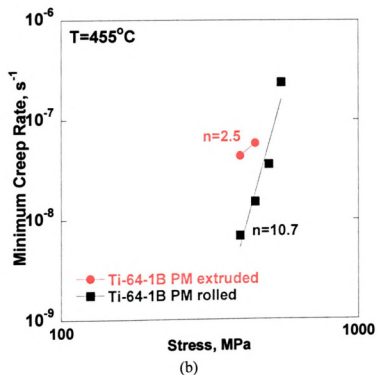
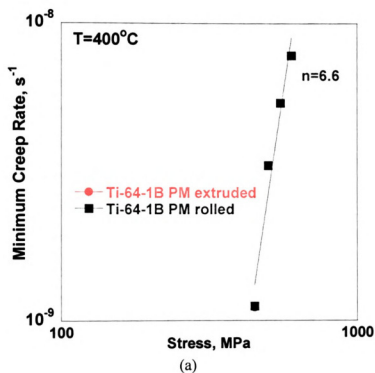


Figure 4.65. Minimum creep rate ($\dot{\epsilon}_{\min}$) versus σ plot for the PM processed Ti-64-1B alloys at (a) 400°C and (b) 455°C illustrating the variation of minimum creep rate with stress. The creep stress exponent (n) values determined from these plots are shown.

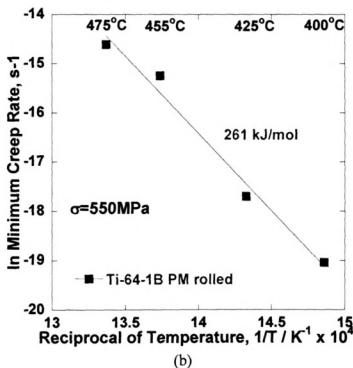
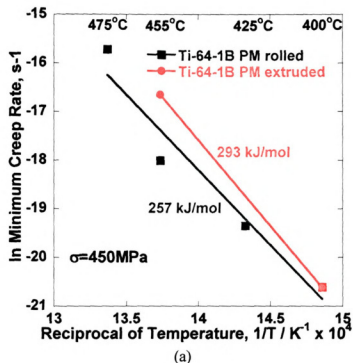


Figure 4.66. $\ln \epsilon_{\min}$ versus reciprocal of temperature for the (a) PM rolled and PM extruded Ti-64-1B alloys at $\sigma = 450 \text{ MPa}$ and for the (b) PM rolled Ti-64-1B alloy at $\sigma = 550 \text{ MPa}$ illustrating the variation of minimum creep rate with temperature. The apparent creep activation energy (Q_{app}) values determined from these plots are shown.

1.2.2 In-situ Creep Behavior

The displacement versus time curve from an *in-situ* tensile-creep experiment is shown in Figure 4.67. Accurate quantitative comparisons were not practical for the conventional and *in-situ* experiments as the *in-situ* experiments were performed in vacuum while the conventional creep experiments were performed in an air environment and the *in-situ* experiments did not utilize an extensometer. Thus, the strain in the heated gage section for the *in-situ* experiments was likely greater than that based upon a uniform strain assumption using the displacement.

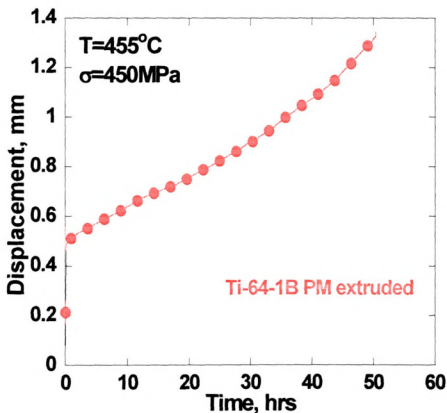


Figure 4.67. Displacement versus time curve for the *in-situ* tensile-creep experiment for the Ti-64-1B PM extruded sample tested at $\sigma=450$ MPa and $T=455^{\circ}\text{C}$.

In-situ creep observations indicated that the TiB whiskers cracked extensively for the PM extruded Ti-64-1B alloy, see Figures 4.68-4.70. In some cases, the TiB whiskers exhibited microcracks at low displacement levels (500 μm) upon loading to the creep stress, and these cracks grew into macrocracks and multiplied with increased creep strain and time. Localized slip in the $\alpha+\beta$ microstructure initiated after the TiB whisker cracking became extensive. Deformation continued via large plasticity in the $\alpha+\beta$ microstructure followed by crack formation in the $\alpha+\beta$ phases before final ductile fracture. The cracks, which always initiated in the TiB whiskers, grew along the loading direction to displacements as large as 3 μm . During the TiB crack growth process, the $\alpha+\beta$ matrix exhibited slip followed by void formation, see Figures 4.70. No slip was observed in the TiB whiskers, and after subtracting the length of the cracks in the TiB whiskers, it was determined that the length of the TiB whiskers did not increase noticeably. Thus it was assumed that little-to-no plastic deformation occurred in the TiB phase. A lower-magnification image of the same area exhibited in Figure 4.70 is shown in Figure 4.71. This indicates the extent of matrix cracking and slip in the α and β phases. SEM images taken from polished gage sections of the deformed creep specimens confirmed that TiB cracking was also evident within the bulk of the material (see Figures 4.72a and b).

Figure 4.68. BSE SEM images for a Ti-64-1B PM extruded sample tested at $T=455^{\circ}\text{C}$, $\sigma=450\text{ MPa}$. The displacements are indicated in “mm” on these images. The loading axis was horizontal in the images. Note that the $\alpha+\beta$ interface was not sliding as seen by tracking the fiducial scratch in the figures.

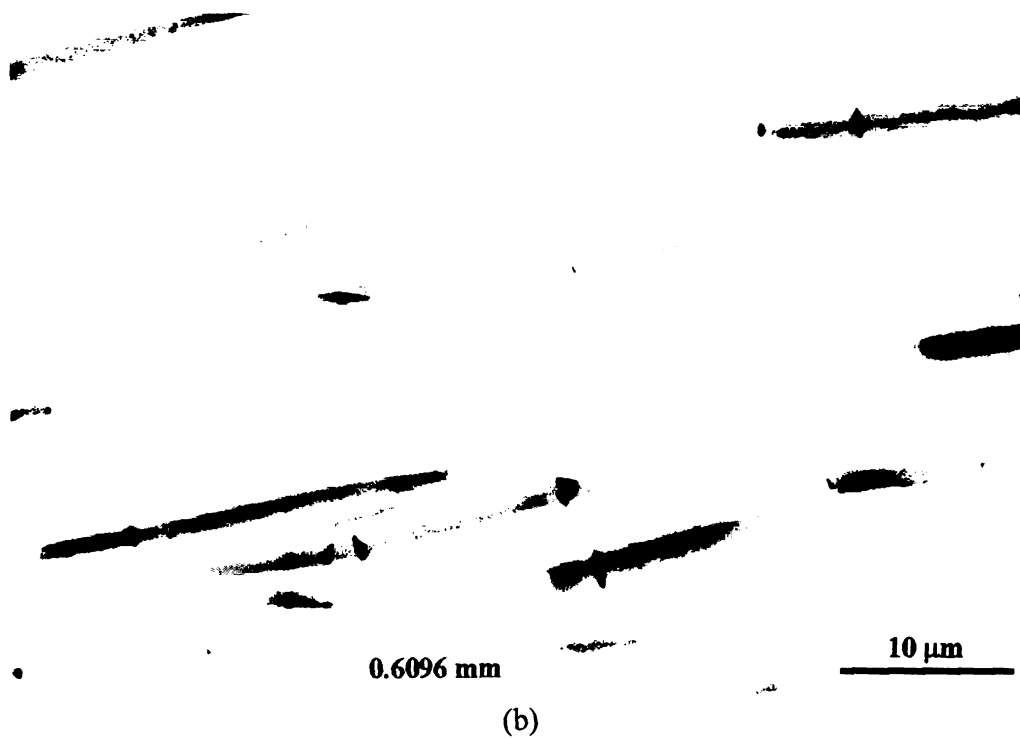
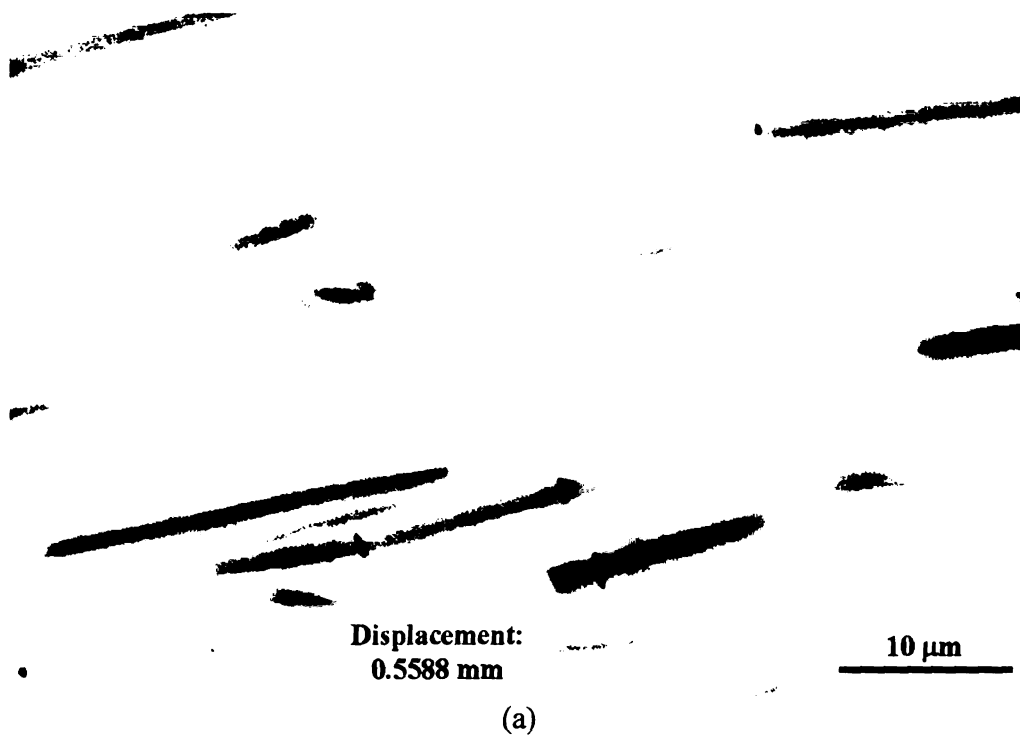
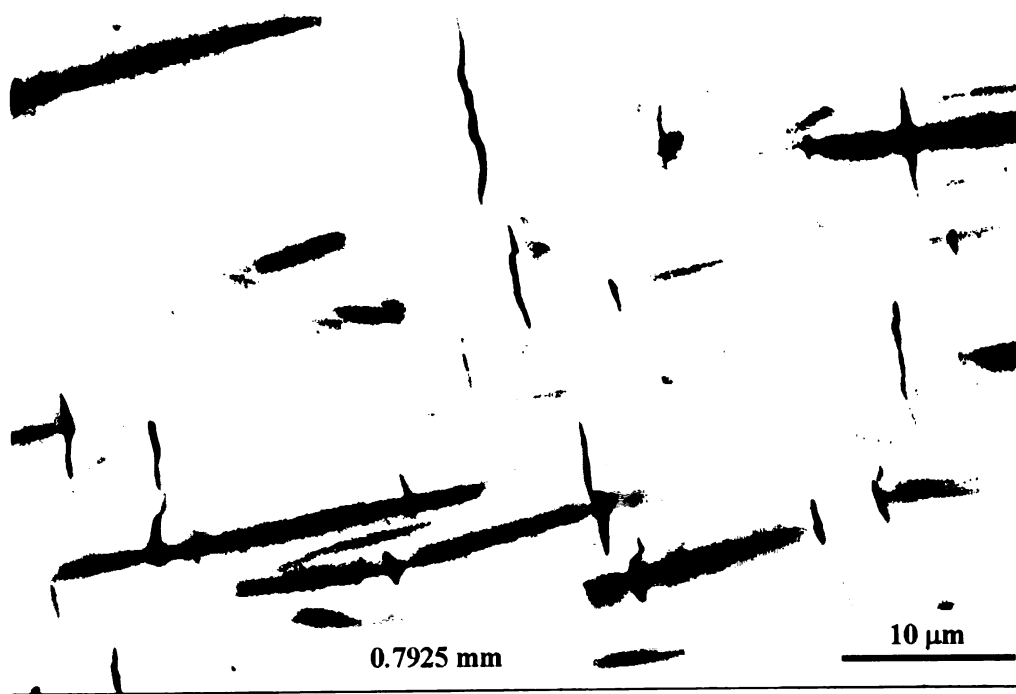
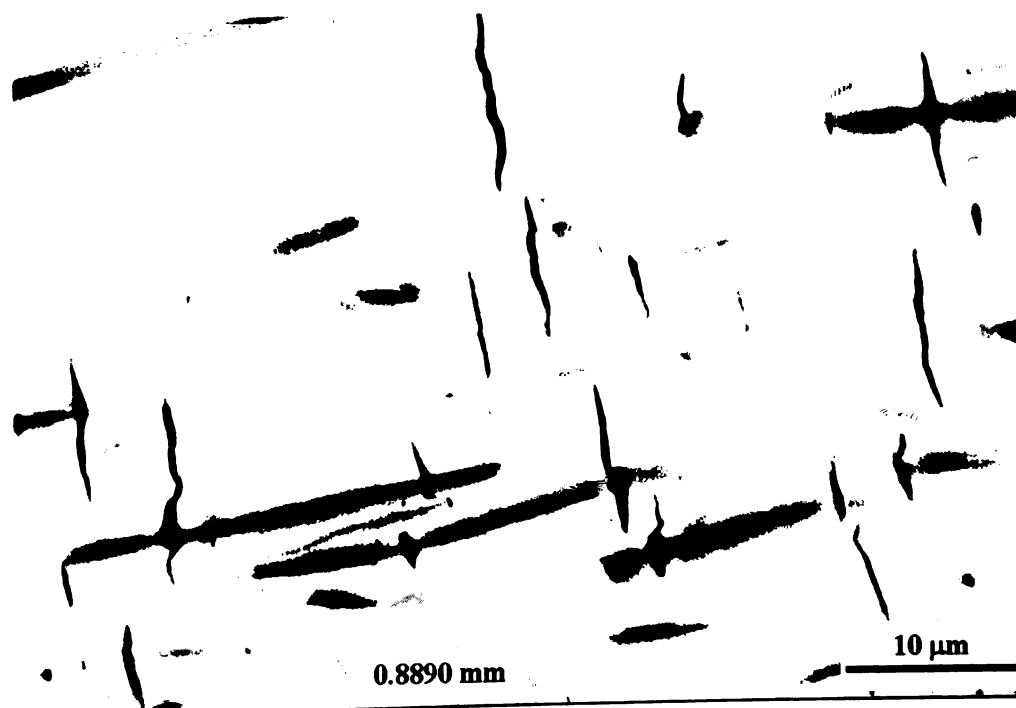


Figure 4.68 (continued)



(c)



(d)

Figure 4.68 (continued)

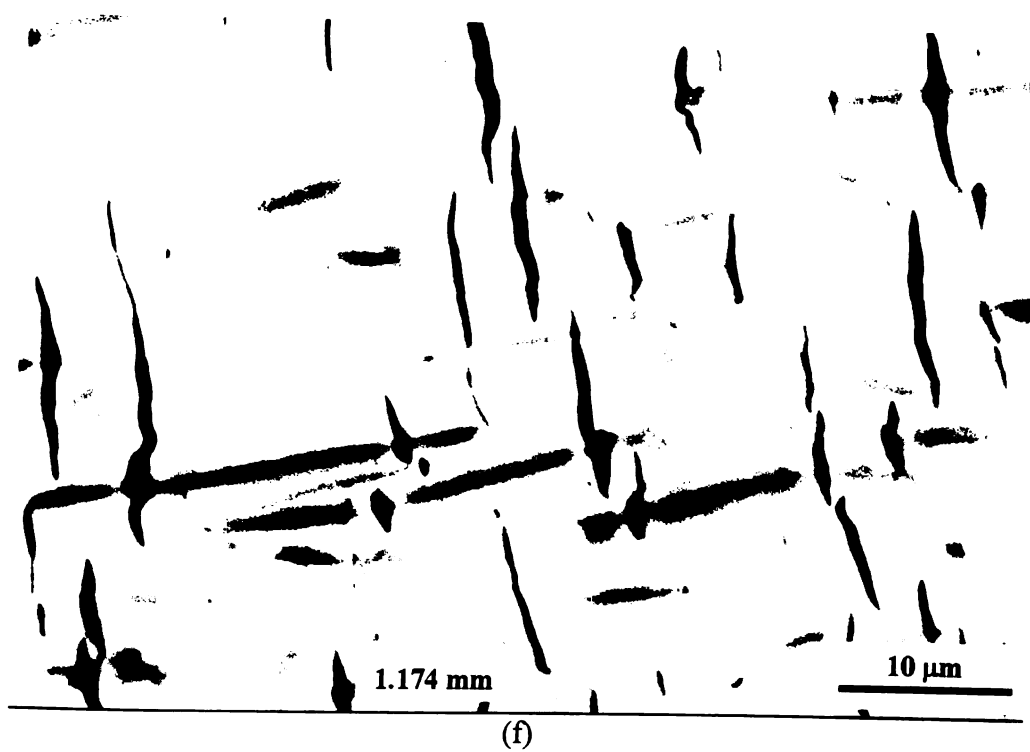
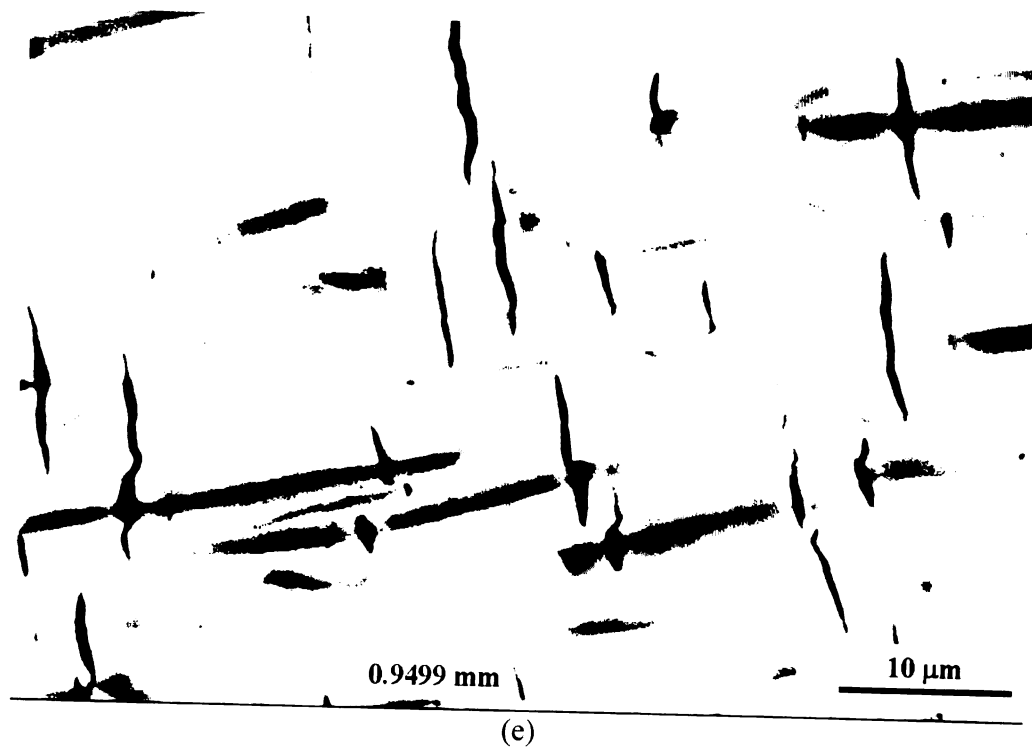


Figure 4.68 (continued)

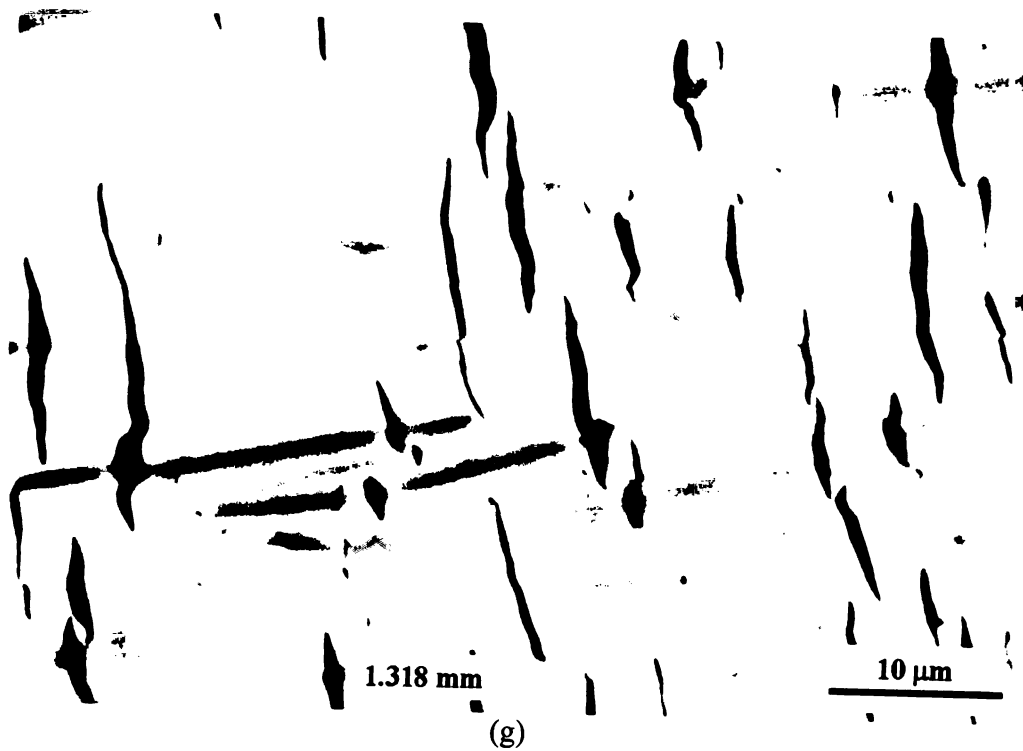


Figure 4.69. BSE SEM images for a PM extruded Ti-64-1B sample tested at $T=455^{\circ}\text{C}$, $\sigma=450\text{ MPa}$. The displacements are indicated in “mm” on these images. The loading axis was horizontal in the images. Note that the TiB whisker oriented perpendicular to the tensile axis did not exhibit cracking.

=455°C
loading
equilibrium

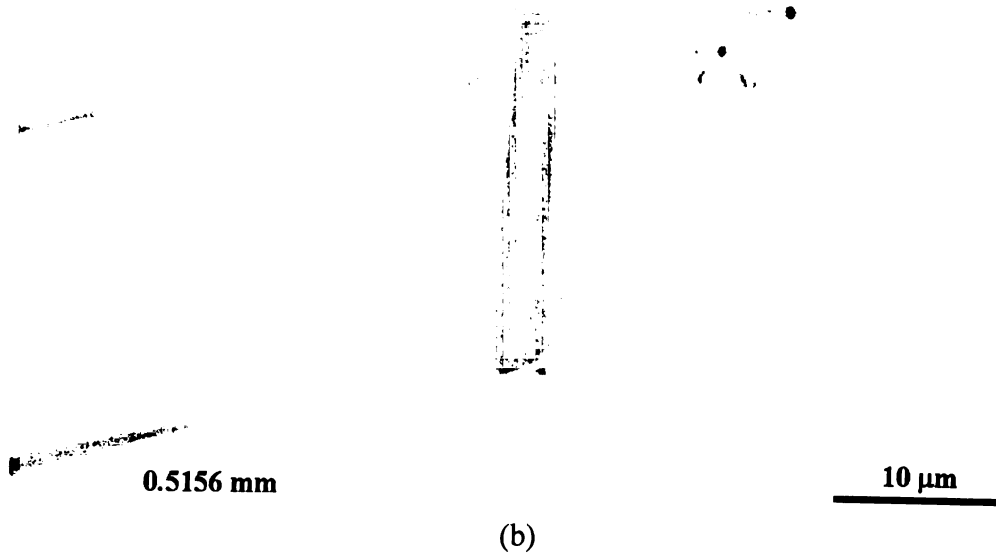
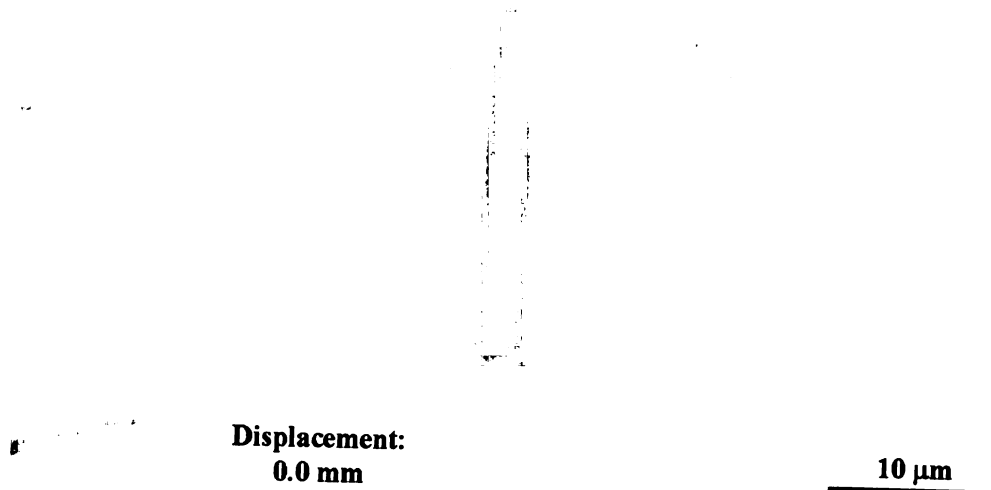
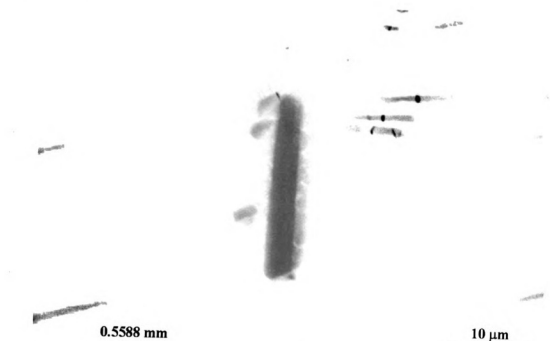


Figure 4.69 (continued)



(c)



(d)

Figure 4.69 (continued)

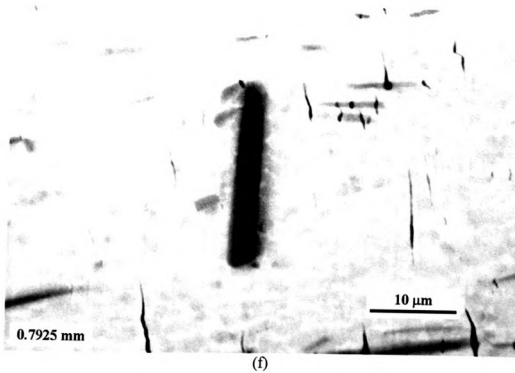
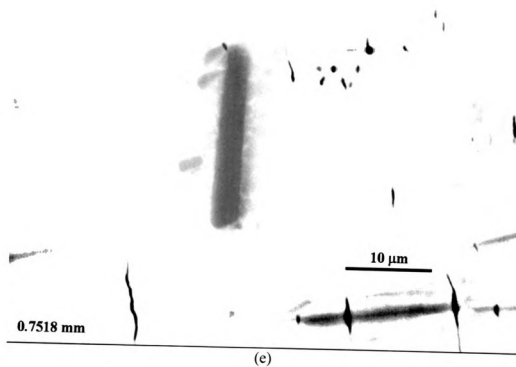


Figure 4.69 (continued)

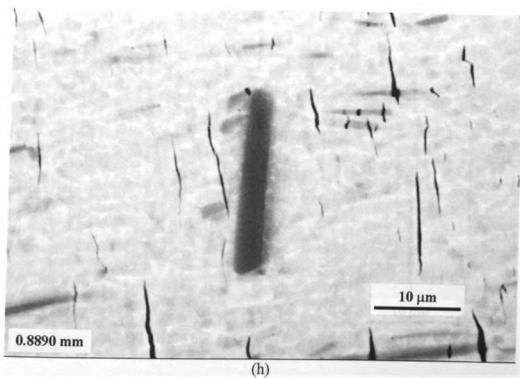
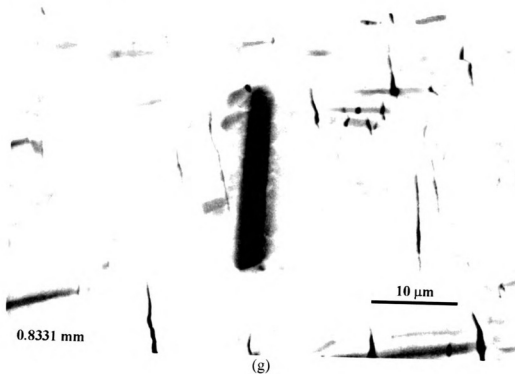


Figure 4.69 (continued)

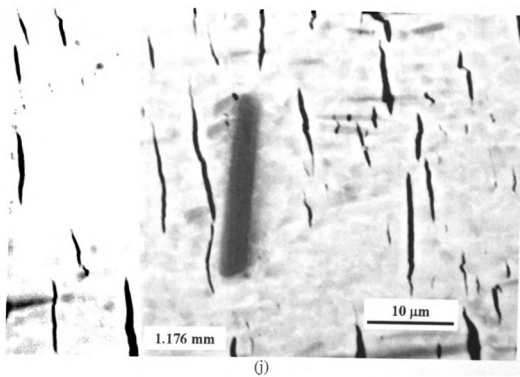
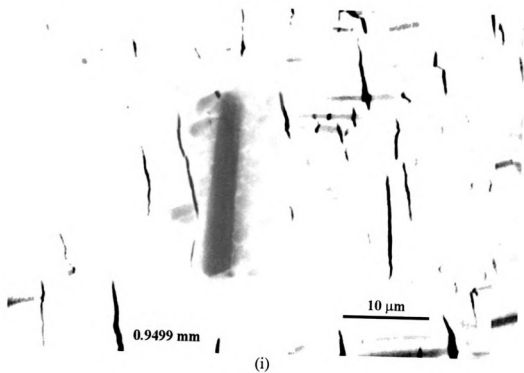


Figure 4.69 (continued)

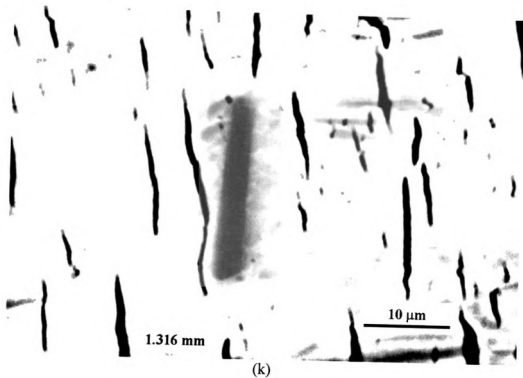


Figure 4.70. BSE SEM images for a Ti-64-1B PM extruded sample tested at $T=455^{\circ}\text{C}$, $\sigma=450\text{ MPa}$. The creep displacements are indicated in “mm” on these images. The loading axis was horizontal in the images.

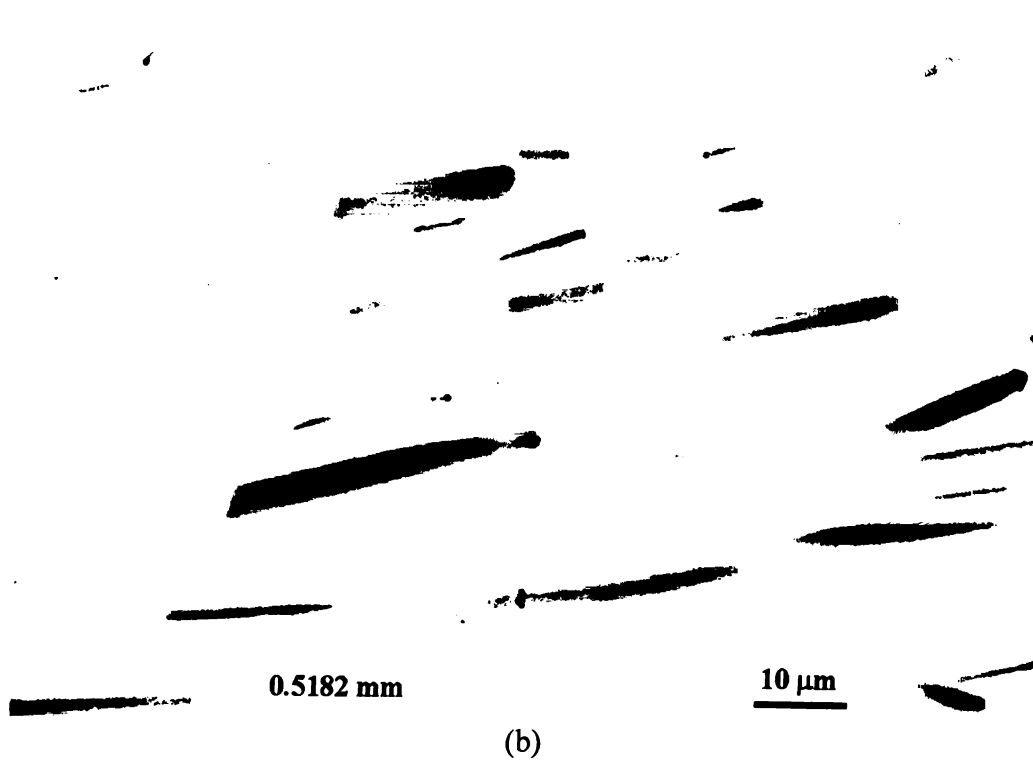
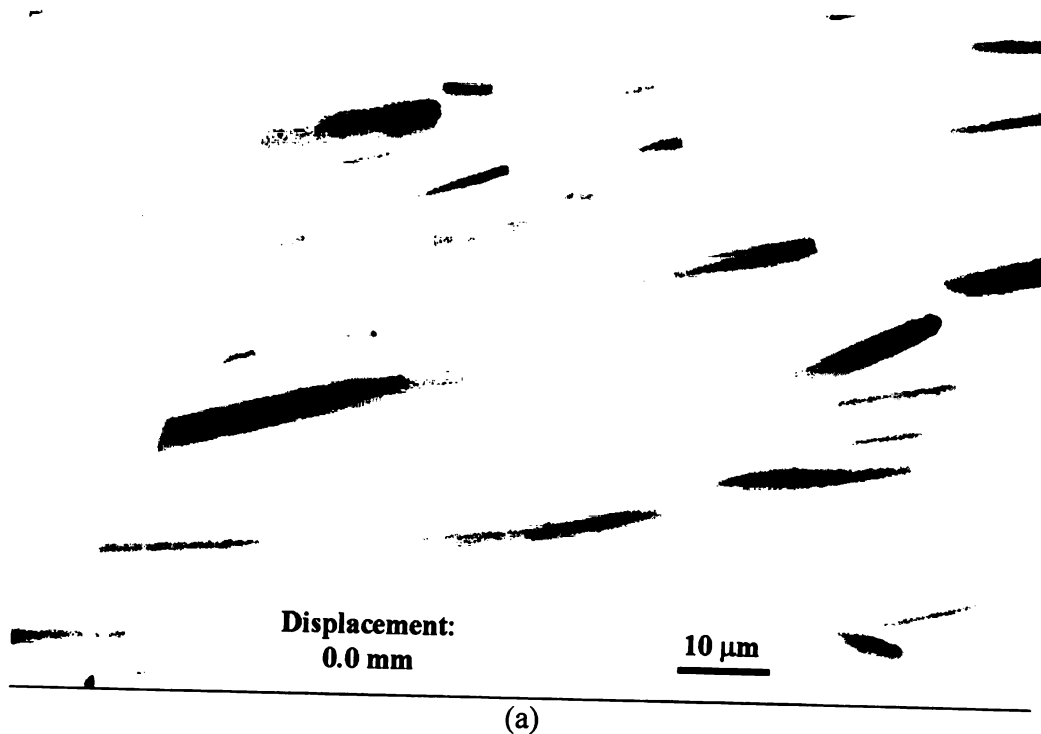


Figure 4.70 (continued)

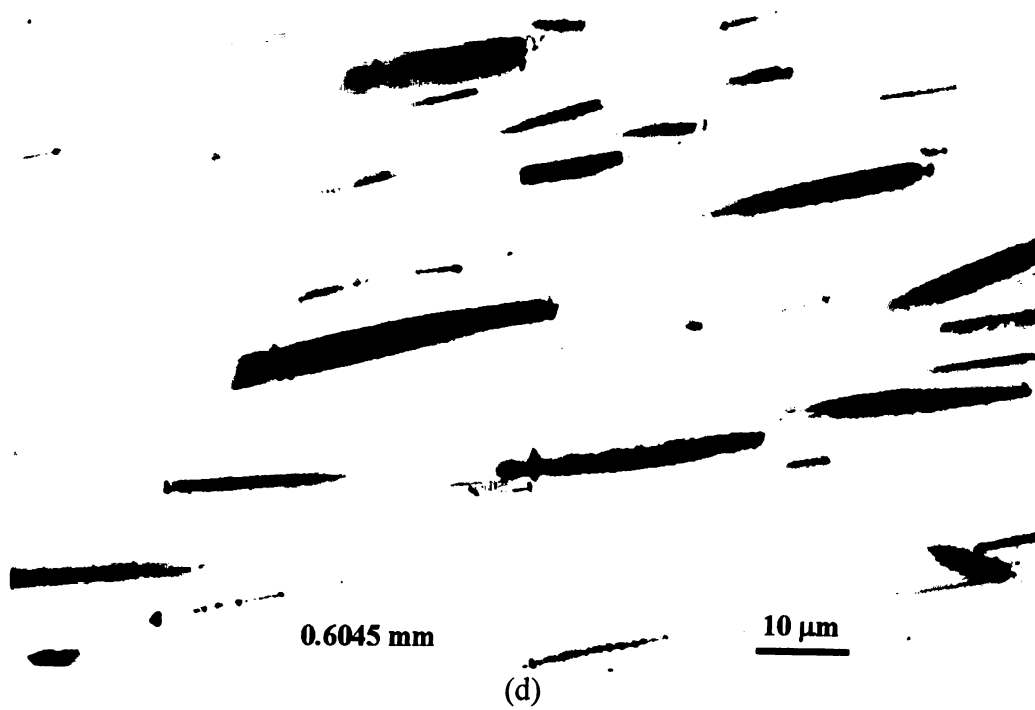
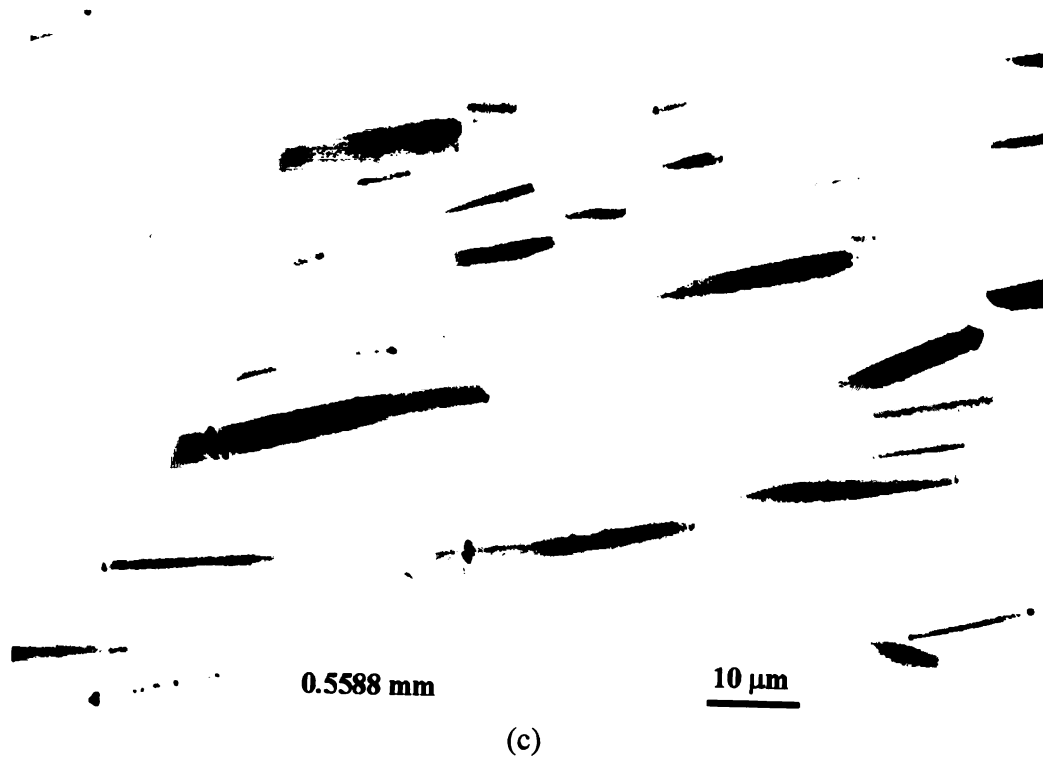


Figure 4.70 (continued)

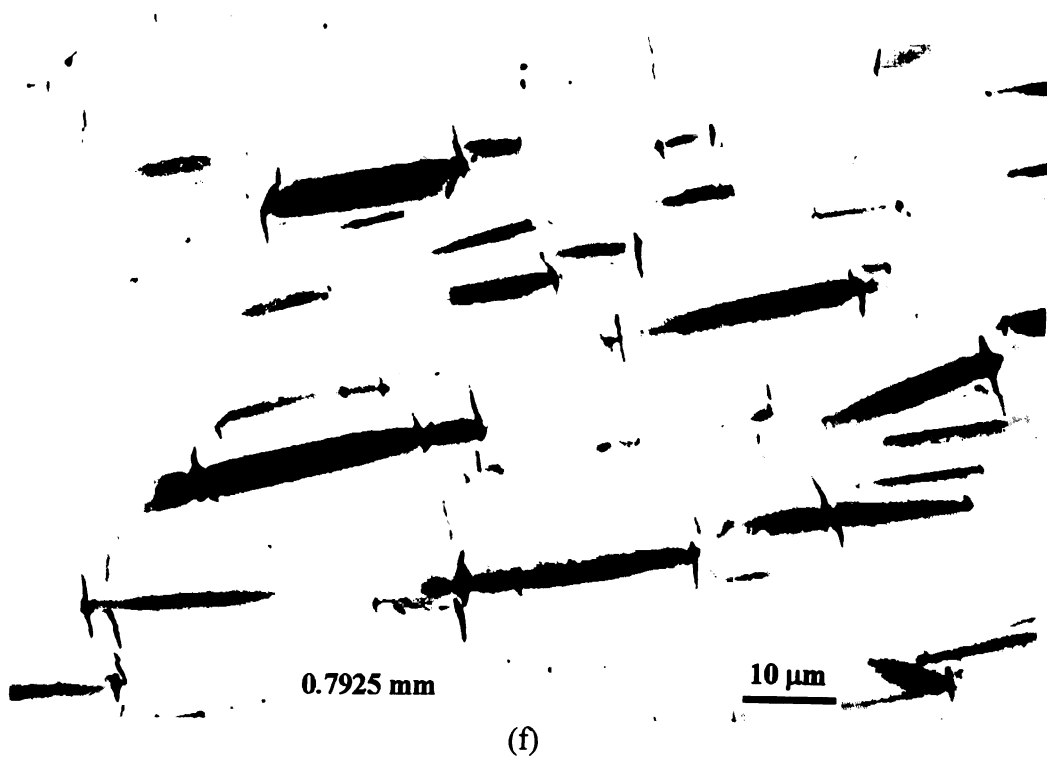
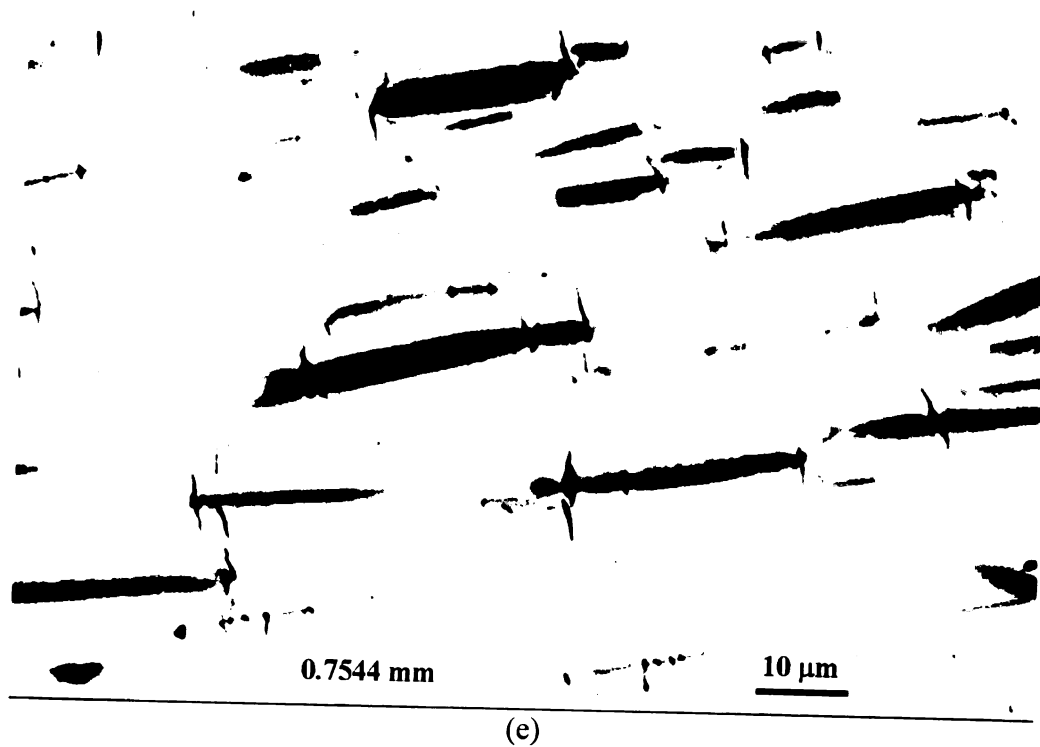
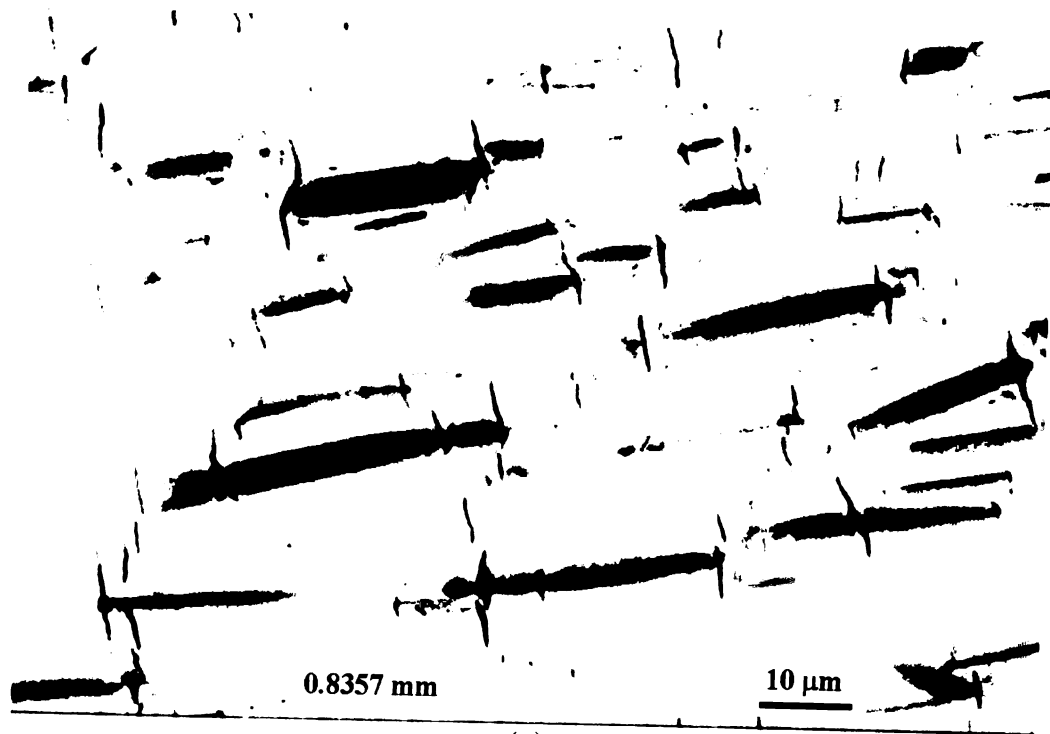
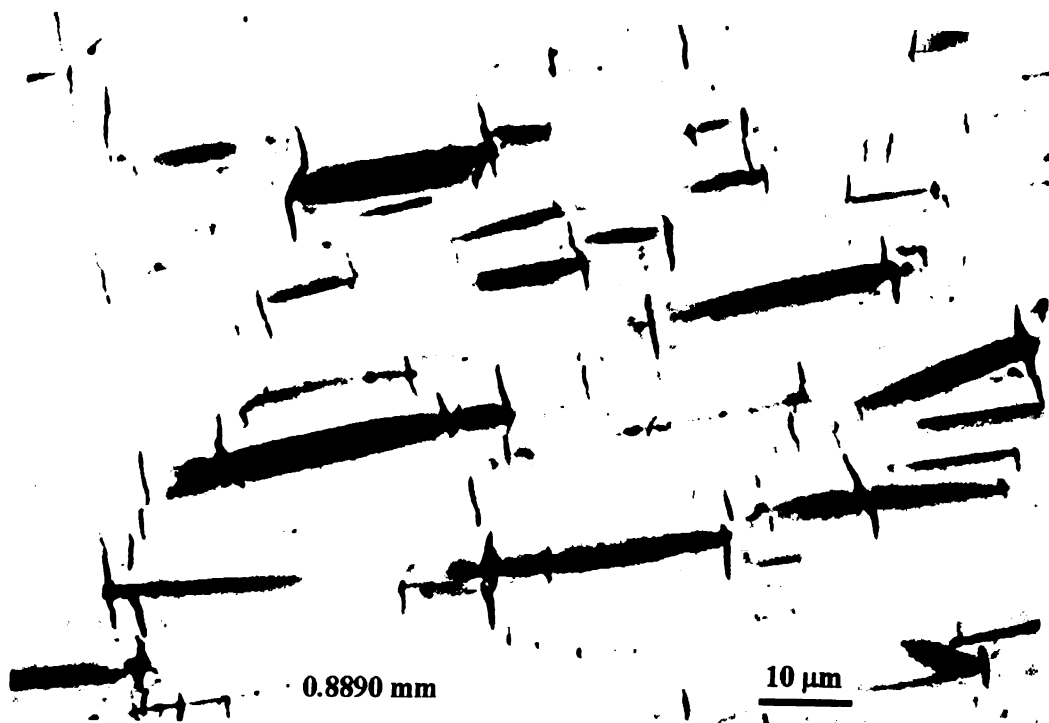


Figure 4.70 (continued)



(g)



(h)

Figure 4.70 (continued)

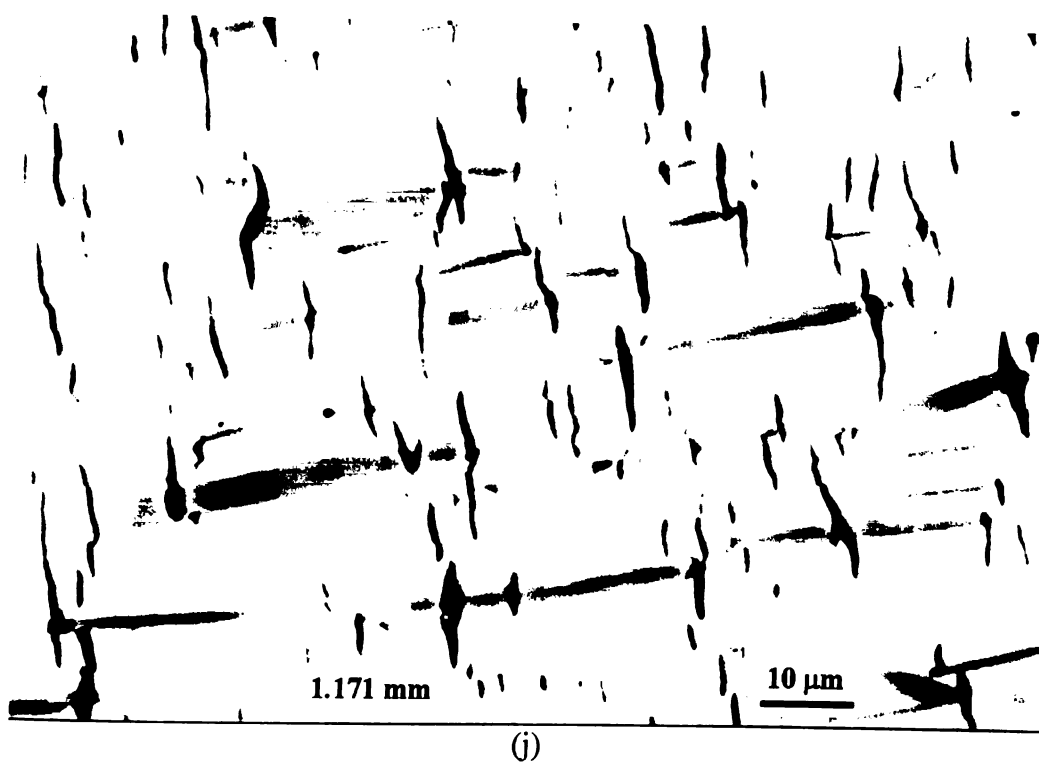
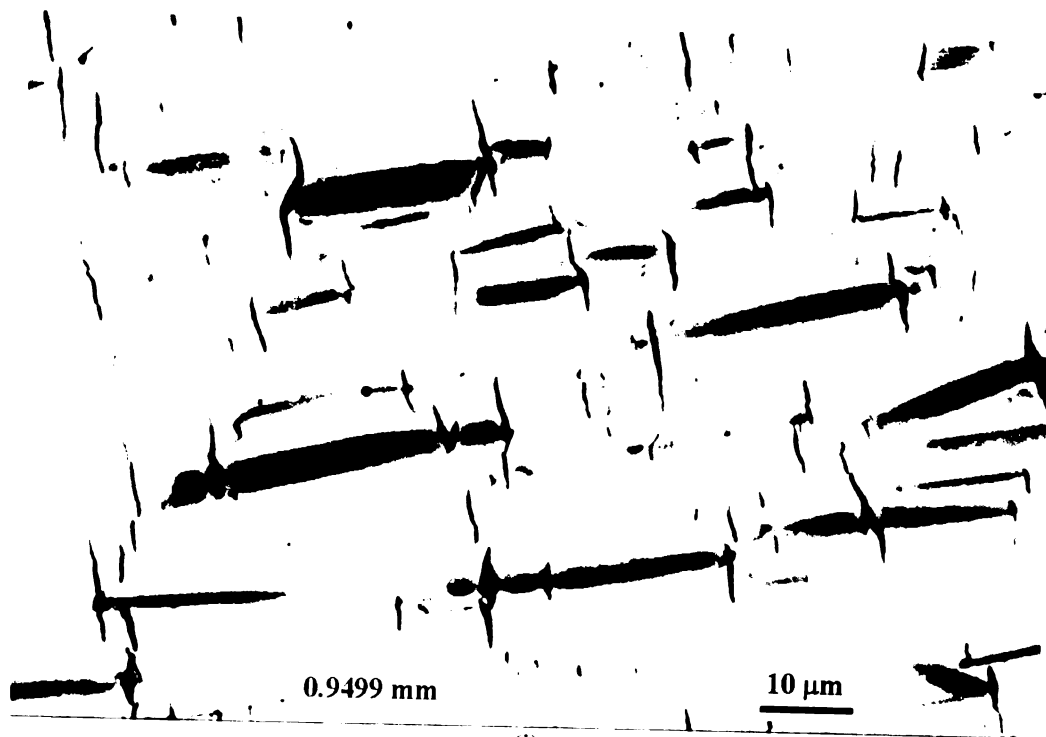


Figure 4.70 (continued)

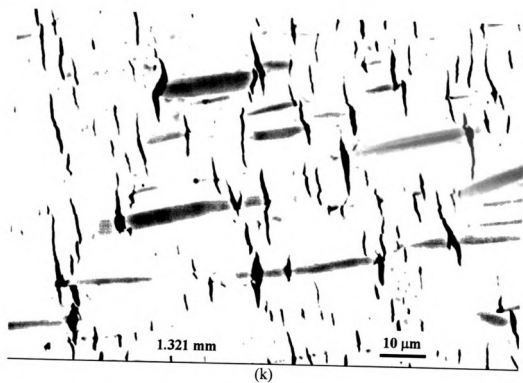
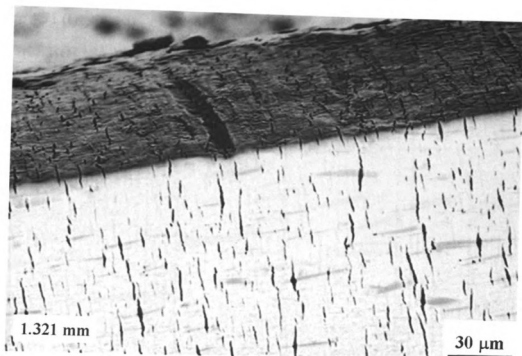
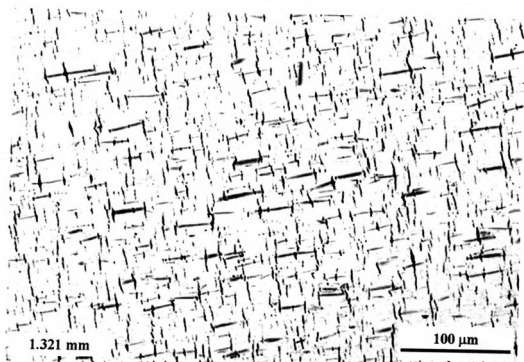


Figure 4.71. Lower-magnification BSE SEM image for the Ti-64-1B PM extruded sample depicted in Figure 48 after the test was completed. (a) Taken from the edge of the sample including the thickness section at 1.323 mm displacement. (b) 250x taken of same area as in above figure at 1.321 mm displacement (c) 500x taken of same area as in above figure at 1.321 mm displacement The loading axis was horizontal in the image.

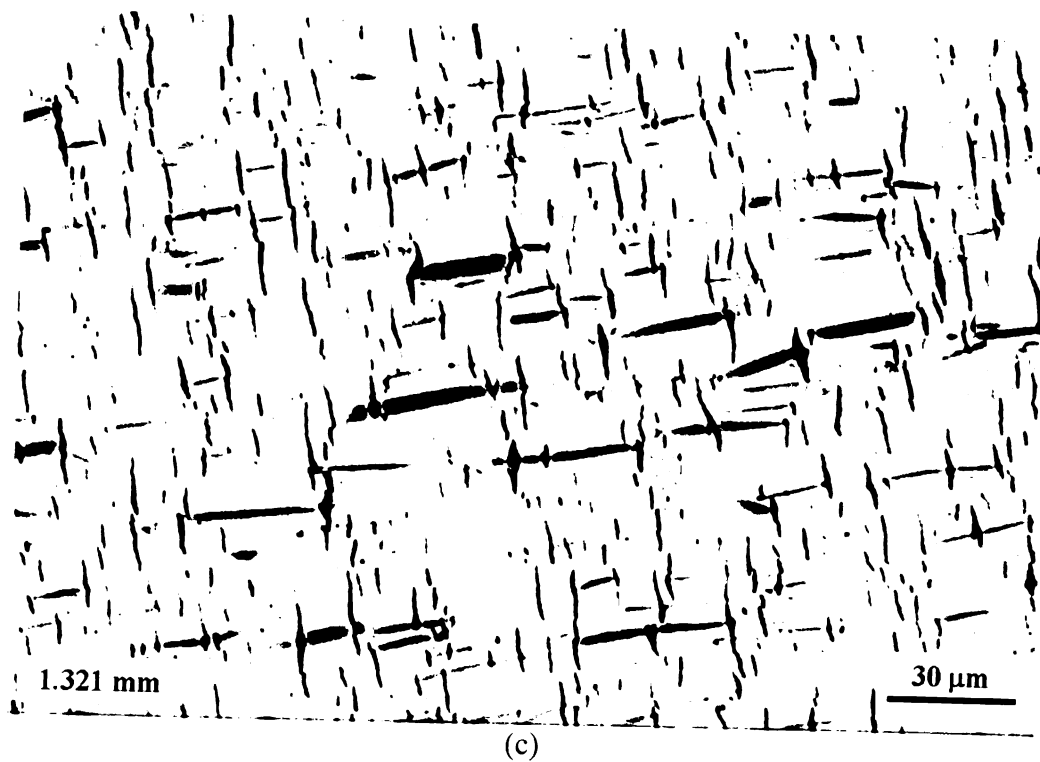


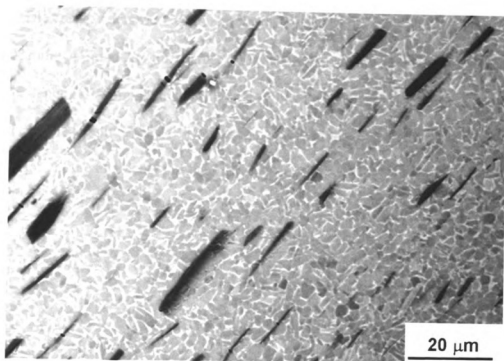
(a)



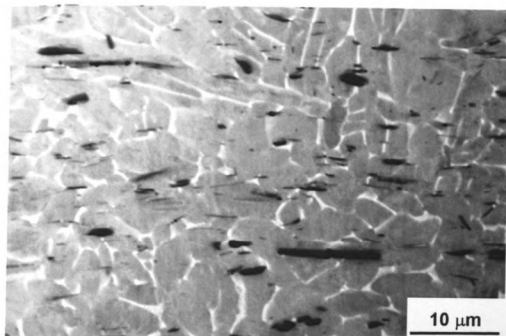
(b)

Figure 4.71 (continued)





(a)



(b)

Figure 4.72. BSE SEM images of the subsurface thickness sections of (a) Ti-6Al-4V PM extruded sample (face) which exhibited 5% creep after undergoing a creep test at $T=455^{\circ}\text{C}/\sigma=400\text{MPa}$ (The loading direction was the same as the TiB orientation) and (b) rolled plate sample after undergoing a creep test at $T=455^{\circ}\text{C}/\sigma=450\text{MPa}$ (The horizontal axis was the loading direction). The samples exhibited TiB whisker cracking.

The crack widths were measured for the cracked TiB whiskers. The crack widths ranged from 0.4 μm to 2.6 μm . Larger crack widths tended to be measured for larger TiB whisker lengths. Normalizing the total length of the crack widths for a given TiB whisker by the length of the TiB whisker indicated that the total crack widths represented from 2-36% of the total length of the TiB whiskers.

2. As-cast Ti-6242S-xB Alloy System

2.1 Creep Properties

Similar to that in the Ti-64-xB alloy system, the creep strain-life behavior of each alloy resembled that for most pure metals and alloys [Evans and Wilshire (1985)]. Some of the creep data obtained are illustrated in Figures 4.73-4.74. The Ti-6242S-1B alloy exhibited lower minimum strain rates than the Ti-6242S base alloy at equivalent applied stresses and creep temperatures, though the minimum creep rates maintained were within a factor of two. In general, the alloys containing 0.1 wt.% B and 0.4 wt.% B exhibited minimum strain rates intermediate to those for the baseline and 1wt.%B alloys, though some exceptions existed for a few stress/temperature combinations. The creep stress exponents (n) calculated using the minimum creep rate versus stress plots shown in Figure 4.73 ranged from 3.4 to 9.3. The apparent activation energy (Q_{app}) values ranged from 292 kJ/mol to 334 kJ/mol at $\sigma=550$ MPa, see Figure 4.74. No phase instability was observed on the post-creep gage sections even after more than 600 hours of creep exposure at 565°C. In such cases the measured volume percents of the TiB, α , and β phases were similar both before and after the experiments.

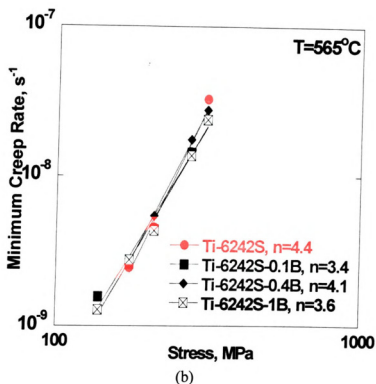
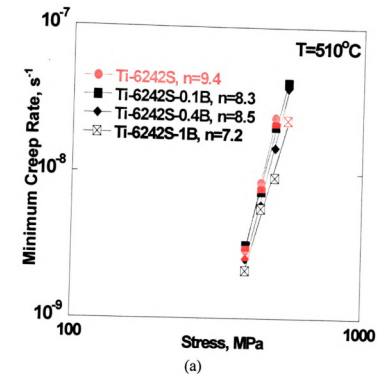


Figure 4.73. Log $\dot{\epsilon}_{\min}$ versus log σ plot at (a) $T=510^{\circ}\text{C}$ and (b) $T=565^{\circ}\text{C}$ illustrating the variation of minimum creep rate with stress. The creep stress exponent (n) values determined from these plots are shown.

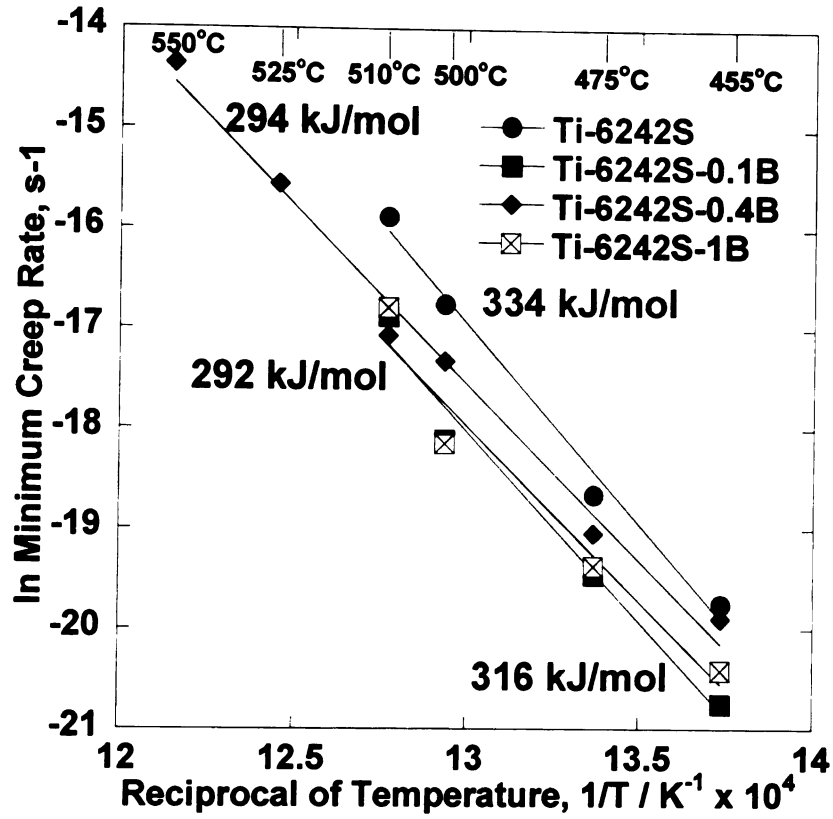
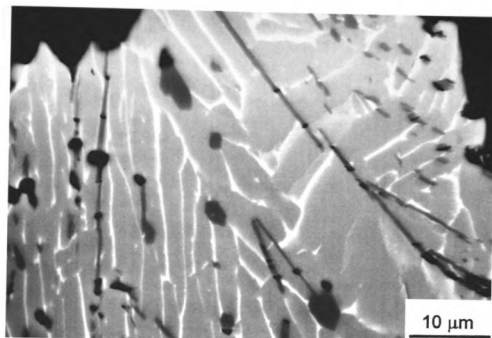
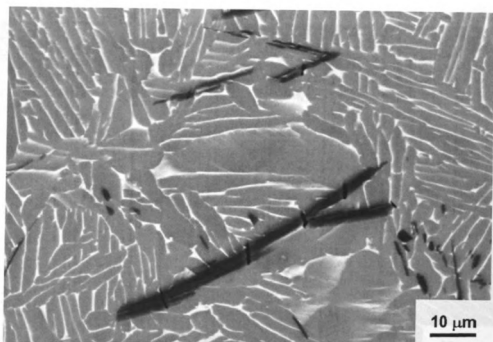


Figure 4.74. $\ln \dot{\epsilon}_{\min}$ versus $10000/T$ curve for the as-cast samples at $\sigma=550$ MPa illustrating the variation of minimum creep rate with temperature. The creep apparent activation energy (Q_{app}) values determined from these plots are shown.

Deformation observations for the samples which underwent the conventional creep experiments in air indicated that extensive TiB whisker cracking was evident, and the extent of the TiB whisker cracking increased with increased creep strain. SEM images taken from polished gage sections of the deformed creep specimens, see Figure 4.75a-b, indicated the extent of the TiB whisker cracking within the bulk of the material. In addition, environmentally-assisted cracking was evident at the edges of the samples where a thin α case layer had developed due to oxidation at the surface, see Figure 4.76a-b. Such cracking occurred preferentially along the α -case layer at the surface which was formed due to oxidation.

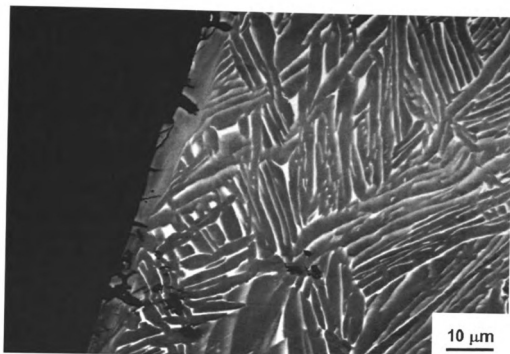


(a)

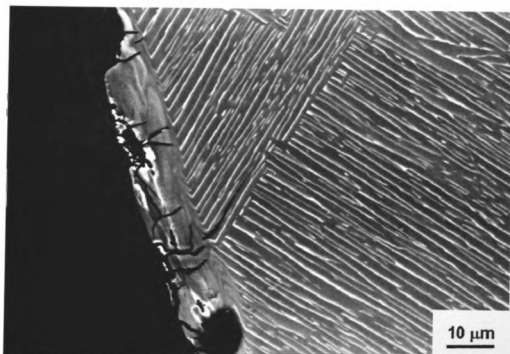


(b)

Figure 4.75. BSE SEM images of the subsurface thickness sections of (a) a Ti-6242S-1B sample which exhibited approximately 1.7% creep after undergoing a load-jump creep test at $T=510^{\circ}\text{C}/\sigma=400\text{-}450\text{-}500\text{-}550\text{MPa}$ and (b) a Ti-6242S-0.4B sample which exhibited approximately 4.0% creep after undergoing a load-jump creep test at $T=510^{\circ}\text{C}/\sigma=400\text{-}450\text{-}500\text{-}550\text{MPa}$. The horizontal axis was the loading direction. The samples exhibited TiB whisker cracking.



(a)



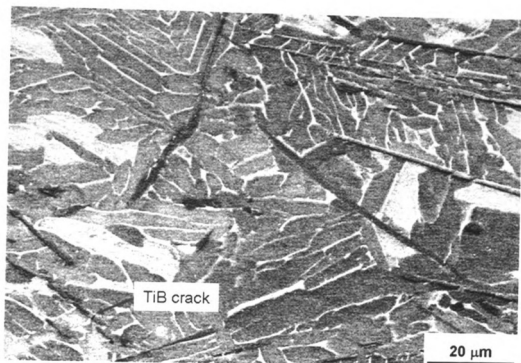
(b)

Figure 4.76. BSE SEM photomicrographs indicating environmentally-affected secondary cracks at the edges of the samples for (a) Ti-6242S-0.1B which exhibited approximately 1.4% creep after undergoing a temperature-jump creep test at $T=475-500^{\circ}\text{C}/\sigma=550\text{MPa}$ and (b) Ti-6242S which exhibited approximately 1.4% creep after undergoing a load-jump creep test at $T=510^{\circ}\text{C}/\sigma=400-450-500\text{MPa}$.

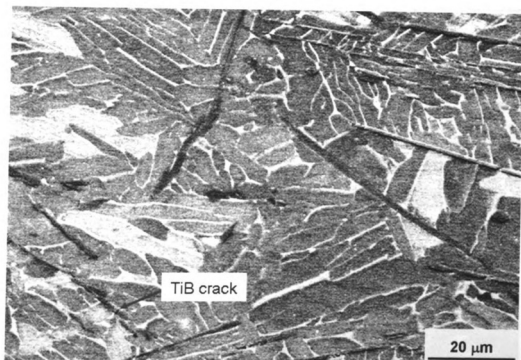
2.2 *In-situ* Creep Behavior

Figure 4.77 illustrates the deformation evolution as a function of displacement during creep at $T=565^{\circ}\text{C}$ as observed on the surface of the *in-situ* tested Ti-6242S-1B sample. The TiB whisker cracks became evident prior to α - and β -phase slip. Figure 4.78 illustrates the fracture surface of this specimen, which exhibited some ductile dimples. It is noted that the specimen failure did not occur across the area imaged in Figure 4.77, thus the surface fracture events were not captured.

Figure 4.77. BSE SEM images for a Ti-6242S-1B sample tested at $T=565^{\circ}\text{C}$. The creep displacements were (a) 0.434 mm (b) 0.523 mm (c) 0.549 mm (d) 0.572 mm (e) 0.617 mm. The loading axis was horizontal in the images. The stress was increased from 310 MPa to 710 MPa at periodic intervals during the test.



(a)



(b)

Figure 4.77 (continued)

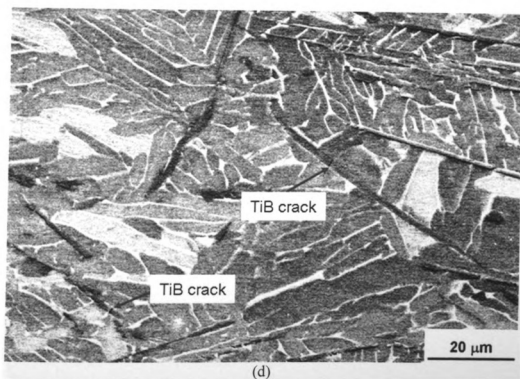
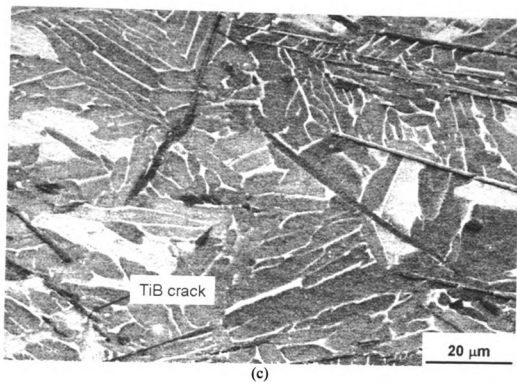
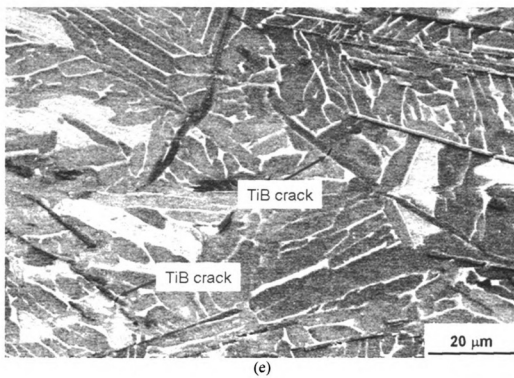


Figure 4.77 (continued)



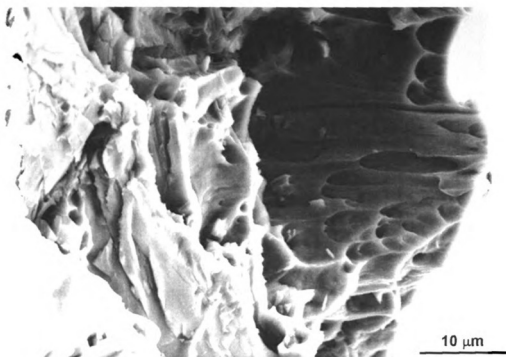


Figure 4.78. SE SEM image of the fracture surface of the *in-situ* creep tested samples.

CHAPTER 5

DISCUSSION

This chapter provides a discussion and detailed analysis of the results presented in Chapter 4, and it is organized to follow the specific aims given at the end of Chapter 1. Discussed first is the effect of B additions and processing on the Ti-64 and Ti-6242S alloy microstructures, followed by microstructure-tensile property relationships. The Harpin-Tsai model is used to model the effect of the TiB phase on the Young's modulus of the composite. The effect of α -phase texture (caused by the extrusion process) on the Young's modulus is then discussed. This model is also used to quantify the α -phase texture weakening due to the addition of the TiB phase in the cast-and-extruded Ti-64-xB alloys. The fatigue behavior of the studied alloys is further evaluated, including a detailed discussion of the fatigue crack initiation, crack propagation and the final failure in relation to the microstructure. The creep deformation mechanisms are suggested from the measured stress exponents and the apparent activation energies as well as the microstructural deformation observations. The effect of B addition and processing on the creep deformation concludes the creep discussion. During the 455°C fatigue experiments, the creep-induced strain was evaluated. These values were compared with the strain in the creep experiments, and fatigue-creep interactions are discussed.

A. Microstructure Discussion

1. Ti-64-xB Alloy System

1.1 As-cast and Cast-and-extruded Alloys

The α -phase morphology depended on the processing route. For processing within the β -phase field, a lenticular α morphology occurs, while for processing (imparting a sufficient amount of deformation) within the α - β phase field, the primary α + β morphology becomes globular (equiaxed) during the subsequent heat treatment in the α + β phase field [Froes (2004)]. Since the cast ingots were cooled from above the β -transus temperature and no thermo-mechanical processing was performed thereafter, the α phase morphology in the as-cast Ti-64-xB alloys was lenticular.

The B-containing as-cast alloys exhibited a greater α -lath width size than as-cast Ti-64. The reason for this is that increasing B contents increase the β -transus temperature of the Ti-64 alloy [Tamirisakandala (2005)]. Thus, B addition results in larger lath sizes due to longer cooling time from the higher β -transus temperature – allowing longer diffusion times and thus larger α -laths. In addition, B is an α stabilizer [Boyer (1994)]. The B stabilized the α phase during the $\beta \rightarrow \alpha$ transformation, and this resulted in a slightly larger α lath width in the as-cast Ti-64-0.1B alloy compared with the Ti-64 alloy. When the B concentration is higher (such as in Ti-64-1B) another mechanism counteracted the α -phase stabilizing effect of B. The TiB phase, which forms along with β in the eutectic reaction at the β -transus temperature, facilitates the nucleation of more α laths [Komizo (2007)]. This results in a reduced α -lath width during the $\beta \rightarrow \alpha$ phase transformation for large β and TiB contents.

The β -phase field extrusion played a significant role in altering the microstructure of the castings. Since the extrusion was performed above the β -transus temperature, the α morphology was lenticular after cooling to RT. A finer grain size resulted from the extrusion procedure. The extrusion procedure also resulted in a significantly refined lath width size and crystallographic texturing, as has been described in Chapter 4. The lath widths for the cast-and-extruded alloys were independent of B content. The significantly finer lath widths for the cast-and-extruded alloys were likely a result of the extrusion procedure followed by faster cooling than that for the as-cast melt. It is possible that the large amount of dislocations produced during the extrusion process might act as effective sites for heterogeneous nucleation of the α -laths during cooling.

The enrichment of V in the β -phase and Al in the α -phase (see microprobe analysis in Table 4.6) was expected since V and Al are strong β -phase and α -phase stabilizers, respectively [Boyer (1994)].

1.2 PM Processed Ti-64-1B Alloys

The PM rolled Ti-64-1B alloy exhibited a duplex microstructure, which contained both lenticular and equiaxed α phase. During the rolling process, the billet surface temperature dropped to 950°C, which is below the β transus temperature (999 $\pm$ 14°C [Allvac technical data sheet]). The rolling process following by the 760°C annealing treatment likely introduced some equiaxed α phase into the alloy microstructure.

In the PM extruded Ti-64-1B alloy, equiaxed α phase was predominant with the β phase decorating the grain boundaries. The α grain diameter (3.6 μ m) was only about half that for the PM rolled Ti-64-1B alloy (7.4 μ m). The processing of the PM extruded

Ti-64-1B alloy was the same as that for the PM extruded Ti-64-1.7B alloy described by Tamirisakandala *et al.* [Tamirisakandala (2005)]. In Tamirisakandala's research, the as-received Ti-64-1B pre-alloyed powder exhibited a martensite microstructure. After blind-die compaction at 1200°C, the alloy exhibited equiaxed α phase. After extrusion at 1100°C the alloy revealed a fine-grained ($\sim 5 \mu\text{m}$) equiaxed α phase which is similar to the microstructure of the PM extruded Ti-64-1B in the current study. The α phase morphology of the PM extruded Ti-64-1B was also similar to the PM Ti-64-2.9B alloy studied by Bhat *et al.* [Bhat (2005)]. The different thermal-mechanical processing routes caused the α phase morphology difference in the PM rolled and PM extruded Ti-64-1B alloys.

Similar to that in the as-cast and cast-and-extruded Ti-64-xB alloys, the β -phase was enriched with V (β stabilizer) and the α -phase was enriched with Al (α stabilizer) in the PM processed Ti-64-1B alloy [Boyer (1994)].

2. As-cast Ti-6242S-xB Alloy System

Similar to the Ti-64-xB alloy system, the α -lath width size increased slightly with increased B content for the Ti-6242S-xB alloy system. This was explained previously to be a result of B, which stabilized the α -phase and thereby increased the β -transus temperature. The microprobe data (see Table 4.7) indicated that the TiB phase acquired some Mo and Zr from the baseline alloy. This may have been the reason why slightly lower TiB-phase volume percents were obtained for the Ti-6242S-xB system compared to the Ti-64-xB system. That is, the addition of Mo and Zr may have impaired the growth of the TiB whiskers by slowing the diffusion kinetics during the TiB-phase formation. The overall volume fraction of the TiB phase would be expected to decrease with increased Mo content, as the addition of Mo has been shown to reduce diffusion-controlled reactions in Ti-alloy systems [Porter (1995)]. For example, comparing a Ti-22Al-24.5Nb-1.5Mo alloy to a Ti-22Al-26Nb alloy, the Mo-containing alloy was shown to exhibit a finer O+BCC structure, which was attributed to the sluggish diffusion kinetics of Mo [Smith (1999)]. The presence of Mo and Zr in the TiB phase is also a cause for the non-stoichiometric Ti and B contents in the TiB phase.

The volume percent of the β -phase in the Ti-6242S-xB alloy system was approximately 1.5 times that for the Ti-64-xB system. This was expected based on the larger amount of β -phase stabilizers (Mo, Zr), and this also occurs when no B was present in these alloys. The grain refinement with increased B content in the Ti-6242S-xB alloy system was similar to that for the Ti-64-xB alloy system, however the starting grain size of the baseline Ti-6242S casting was more than three times smaller than that for the Ti-64 casting [Tamirisakandala (2005)]. Overall, in terms of the microstructural

parameters, the main difference between the Ti-6242S-xB and Ti-64-xB systems is that the TiB phase contained approximately 1 wt.% Mo and 1 wt.% of Zr.

B. Texture Discussion

1. As-cast and Cast-and-extruded Ti-64-xB Alloys

For the as-cast alloys, the TiB phase was not strongly textured. This is in agreement with previous observations by Ivasishin *et al.* [Ivasishin (2008)] for an as-cast eutectic Ti-64-1.55B alloy. The extrusion procedure aligned the TiB whiskers in the extrusion direction with [010] aligned parallel to the extrusion direction, see Figure 4.2. This is in agreement with the TEM observations indicating the growth direction of the TiB whiskers to be [010] [Lu (2001), Kooi (2003)]. In addition, direct extrusion of as-cast eutectic Ti-64-1.55B in the β -phase field aligned a majority of the TiB along the extrusion direction [Ivasishin (2008)]. The high aspect ratios of the TiB phase are due to strong anisotropy in growth rate, with the fastest growth in the [010] axial direction of rods and the long dimensions of plates. Thermomechanical processing in the β -phase field allows reasonably easy plastic flow and facilitates rigid body rotation of intermetallic TiB causing alignment along the direction of flow [Schuh (2001)].

In addition, the α -phase became strongly textured during the extrusion procedure. This texture was similar to that resulting from hot rolling of Ti-64 rods [Bieler (2005), Zeng (2005)]. During the β -phase field extrusion, it is expected that the β phase will preferentially align with the $\{110\}$ planes parallel to the extrusion direction as the β phase has 12 slip systems associated with $\{110\}$ family of planes. It is noted that a strong (110) texture has been found in cold-drawn α -Fe (bcc) [Van Acker (1996), Read (1997)]. The $\{110\}$ planes are located either 0° , 60° , or 90° apart in the cubic system. The Burgers orientation relationship between α and β is $(110)\beta // (0002)\alpha$; $[\bar{1}\bar{1}1]\beta // [1\bar{1}20]\alpha$ [Burgers (1934)]. Following transformation to the α phase, the texture

of the β phase would be expected to be inherited by the α phase in a manner consistent with the Burgers relationship as has been observed previously for Ti-64 [Vreeling (2002)]. Thus after the transformation to the α phase, which occurred during the cooldown from the β -phase field, the basal plane normal of the α phase is expected to be either 0° , 60° , or 90° from the extrusion direction, which is in agreement with the experimental observations. In addition, the largest peak for the retained β -phase was for the (110) plane. This has also been observed in previous studies of Ti-64 [Bieler (2005), Zeng (2005)]. Based on the orientation relationship and the symmetries of the cubic and hexagonal structures, a total of 12 crystallographically distinct variants of the α phase can result from the transformation of a single β grain. Variant selection would dictate how much basal plane α is 0° , 10° , 60° , or 90° oriented. The hcp-to-bcc phase transformation in Ti has been observed *in-situ* by EBSD [Seward (2004)] and by neutron diffraction techniques [Lonardelli (2007)]. Lonardelli *et al.* [Lonardelli (2007)] studied the texture evolution of $\beta \rightarrow \alpha$ phase transformation in Ti-64 alloy by *in-situ* neutron diffraction and suggested that the growth of α domains is controlled by high-temperature α orientations inherited from the β grains with Burgers orientation relation.

The XRD pole figures, Figure 4.12, suggested that the basal plane was predominately oriented perpendicular to the extrusion axis for each cast-and-extruded alloy. In the XRD 2θ scans (Figure 4.11), the ratio between the (0002) plane and the (10-10) plane decreased from 2.6(Ti-64) $\rightarrow$ 2.1(Ti-64-0.1B) $\rightarrow$ 1.1(Ti-64-1B) as the B content increased in the Ti-64-xB alloy system, indicating that the greatest fraction of (0002) planes oriented perpendicular to the extrusion axis were for the Ti-64 cast-and-extruded alloy, see Figure 5.1. In agreement with this study, B addition also has been

reported to weaken the texture of a Ti-64 solidified ingot [Roy (2008)]. It is unclear why B weakened the texture, and this may be a topic of future work.

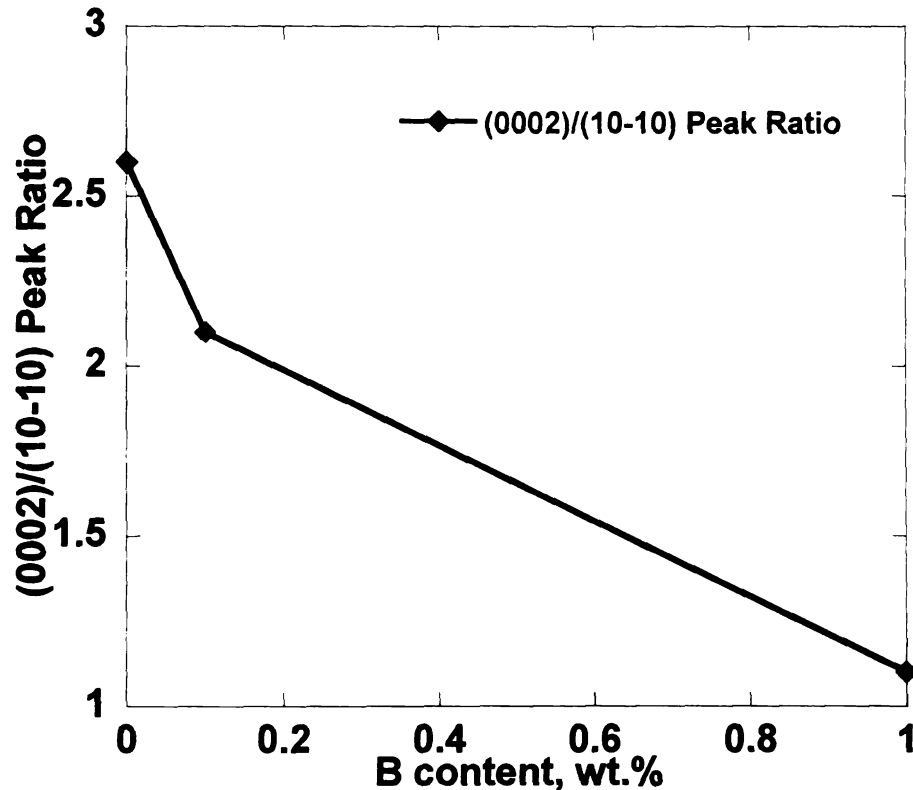


Figure 5.1. (0002)/(10-10) peak ratio vs. B content for the Ti-64-xB cast-and-extruded alloys. The decrease of this ratio indicates the weakening of the (0002) texture in the longitudinal direction.

2. PM Processed Ti-64-xB Alloys

In general, crystallographic textures depend on the degree, the mode, the temperature of deformation and the subsequent annealing process. Usually, four temperature regions are distinguished for uniaxial rolling of the Ti-64 alloy [Peters (2003)]: below 900°C, primarily basal-type textures form; at temperatures between 900°C and 930°C, no strong textures are usually formed; at temperatures just below the β -

transus temperature, the basal component of the texture disappears (leaving only the transverse component); above the β -transus temperature, a classic 110 cubic extrusion texture forms. The PM rolled Ti-64-1B alloy in this study underwent uniaxial rolling (3 rounds, 8 passes for each round) above the β -transus temperature (temperature dropped below the β -transus temperature during the rolling process, as described in Chapter 2) followed by annealing at 760°C. However, neither the EBSD (see Figure 4.10) nor the XRD analysis (see Figure 4.13) indicated a pronounced α -phase texture. In agreement, the rolling process did not cause anisotropy with respect to the tensile properties.

However, EBSD analysis suggested that texture in the TiB phase was developed during the rolling process (see Figures 4.10d and e). The longitudinal axis of the TiB phase ([010] direction) was predominantly oriented parallel to the rolling plane. This was supported by the BSE images on the transverse and longitudinal sections of the PM rolled Ti-64-1B plate (see Figures 4.3a, b, and c). The longitudinal sections exhibited more TiB phase ends while the transverse sections exhibited more of the entire lengths of the TiB-phase whiskers.

In the PM extruded Ti-64-1B alloy, the extrusion process also aligned the TiB phase in the extrusion direction with [010] aligned parallel to the extrusion direction, see Figure 4.3d. Thermomechanical processing in the β -phase field allows reasonably easy plastic flow and facilitates rigid body rotation of the intermetallic TiB causing alignment along the direction of flow [Schuh (2001)]. The alignment of the TiB phase was also observed for the Ti-64-1.7B using the same PM processing conditions [Tamirisakandala (2005)].

Similar to the cast-and-extruded Ti-64-xB alloys, the α -phase in the PM extruded Ti-64-1B alloy became strongly textured during the extrusion procedure. The (0002) plane of the α -phase was predominantly perpendicular to the extrusion axis in the Ti-64-1B PM extruded alloy, which is indicated in Figure 4.9 (EBSD analysis) and Figure 4.13d (XRD analysis). The largest peak for the retained β -phase in the PM extruded Ti-64-1B alloy was also the (110) plane. The mechanism of the α -phase texture development in the PM extruded Ti-64-1B alloy is the same as in the cast-and-extruded Ti-64-xB alloys, which was described previously in this chapter. The peak ratio between the (0002) and the (10-10) planes was higher in the PM extruded Ti-64-1B alloy (3.4) than in the cast-and-extruded Ti-64-1B alloy (1.1), see Figures 4.11f and 4.13d. This indicates that the (0002) texture of the α phase in the PM extruded Ti-64-1B was stronger than that in the cast-and-extruded Ti-64-1B alloy. The reason of this phenomenon is unclear and this may be a topic of future work.

C. Tensile Behavior Discussion

1. Ti-64-xB Alloy System

1.1 As-cast Alloys

From the tensile properties of the as-cast Ti-64-xB alloys shown in Tables 4.8 and 4.9, and Figure 4.14, it is clear that B addition increased the E values and tensile strength of the Ti-64 alloy at 455°C and also at RT. Early work on Ti-64-1.6B alloy concluded that the dominant strengthening mechanism is from load sharing by TiB whiskers [Tamirisakandala (2003)], but the B content in that alloy was much higher than in the current study. Sen *et al.* [Sen (2007)] studied the relationship between YS and prior- β grain size for the as-cast Ti-64-xB (x=0, 0.05, 0.1, and 0.4) alloys and suggested that the Hall-Petch mechanism can be dominant for the Ti-64-xB alloys with low B content. So the strengthening effect in as-cast Ti-64-xB alloys can come from both the load sharing of the TiB phase and grain refinement in the $\alpha+\beta$ microstructure.

Ti-64-0.1B alloy exhibited the highest ϵ_f value (11.8%) of all the as-cast alloys. This was due to the grain refinement effect by B addition and it improved the workability of the Ti-64 alloy [Tamirisakandala (2006)]. However, the ϵ_f value for the as-cast Ti-64-1B alloy was 2.5%. Obviously any expected increase of ϵ_f through grain refinement was offset by the larger volume of brittle TiB whiskers in the as-cast Ti-64-1B alloy. It has been found by Sen *et al.* [Sen (2007)] that the as-cast Ti-64-0.05B alloy exhibited a RT ϵ_f value (8.5%) less than that for the Ti-64-0.1B alloy in the current study. Thus 0.1B may be the optimal B content from a ductility point of view.

Reducing the α -colony size through B addition also played an important role on the YS and ϵ_f values. Lutjering and Williams [Lutjering (2007)] summarized the correlation between microstructural parameters and mechanical properties of $\alpha+\beta$ alloys. They found that smaller α -colony and α -lath width sizes increase the YS and ϵ_f values. Since the α -colony size of the as-cast Ti-64-0.1B and Ti-64-1B alloys are finer than that for the as-cast Ti-64 (see Figure 5.2), it is expected that these alloys would exhibit greater YS values than Ti-64 and thus α -colony refinement would have been expected to contributed to this observed strengthening behavior. The α -lath width of the as-cast Ti-64-xB alloys was quite stable and independent of B concentration, so this factor was not expected to have significantly affected the tensile properties.

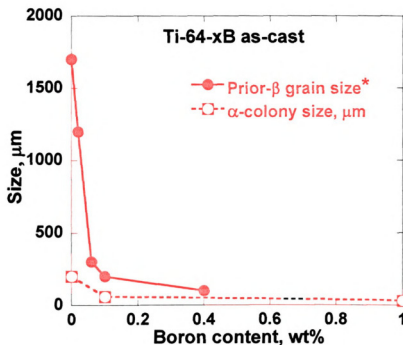


Figure 5.2. Effect of B addition on the prior- β grain size and α -colony size of the as-cast Ti-64 alloy. [Tamirisakandala (2005)]

1.2 Cast-and-extruded Alloys

Processing played a significant role in altering the tensile strength of the alloys. The cast-and-extruded alloys exhibited significantly greater tensile strengths than the as-cast alloys. The tensile strength of the cast-and-extruded Ti-64 alloy was similar to that for wrought solution-treated and annealed Ti-64 [Boyer (1994)]. Unlike that for the as-cast alloys, the B-content did not significantly alter the tensile strengths of the cast-and-extruded alloys at 455°C and RT, see Tables 4.8 and 4.9. This is believed to have been a result of the α -phase texture in the cast-and-extruded alloys. As the majority the microstructure consisted of the α -phase, the texture of the α -phase is an important microstructural parameter for the mechanical properties. If the basal plane is either normal or parallel to the extrusion/tensile axis, then slip along the basal plane is prevented. Thus this represents a “hard orientation”.

As mentioned earlier in this chapter, smaller α -colony size and α -lath width has been shown to increase YS and ϵ_f values [Lutjering (2007)]. The α -colony size for the cast-and-extruded Ti-64-xB alloys was much smaller (by over an order of magnitude) than that for the as-cast Ti-64-xB alloys (see Figures 5.2 and 5.3). The α -lath width for the cast-and-extruded alloys was four times finer than that for the as-cast alloys, see Figure 5.4. These two microstructural features contributed to the higher strength for cast-and-extruded Ti-64-xB alloys compared with the as-cast Ti-64-xB alloys.

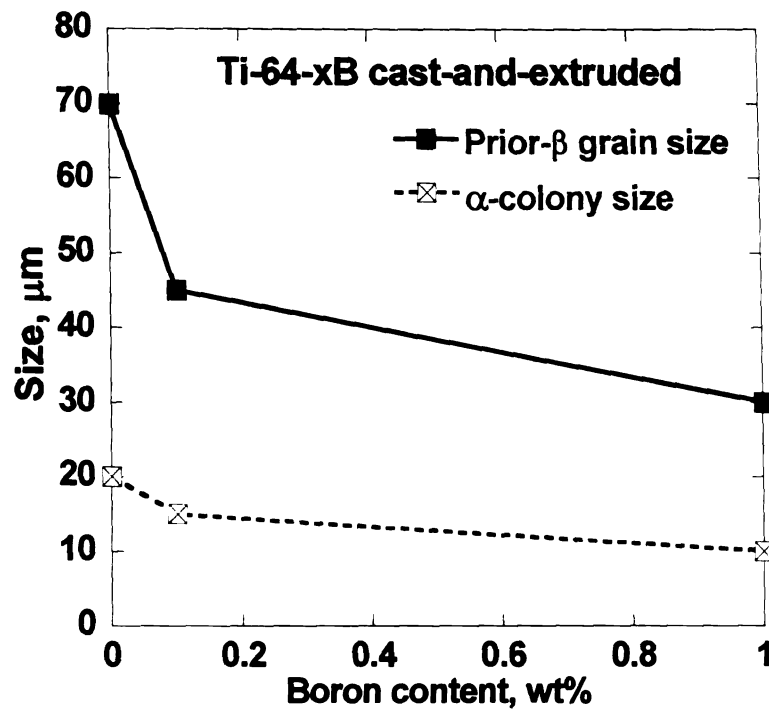
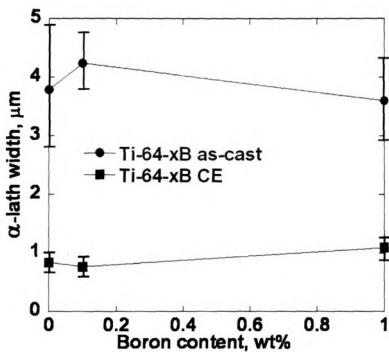
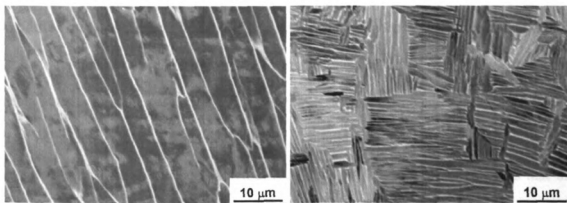


Figure 5.3. Effect of B addition on the prior- β grain size and α colony size of cast-and-extruded Ti-64 alloy.



(a)



(b)

(c)

Figure 5.4. Comparison of α lath width between as-cast and cast-and-extruded Ti-64-xB alloys: (a) α -lath width versus B-content, (b) BSE SEM image of as-cast Ti-64 alloy, and (c) BSE SEM image of cast-and-extruded Ti-64 alloy.

The XRD 2θ plots (Figure 4.11) suggest that the greatest fraction of basal planes oriented perpendicular to the tensile axis were for the Ti-64 cast-and-extruded alloy (the peak ratio between (0002) and (10-10) plane is the highest for the Ti-64 cast-and-extruded alloy). The XRD pole figures (Figure 4.12) also indicates that the Ti-64 cast-and-extruded alloy is more strongly textured than the Ti-64-1B cast-and-extruded alloy. In other words, B addition weakened the texture in the cast-and-extruded Ti-64 alloy. This may be the reason why the B-modified alloys exhibited tensile strengths similar to that for the Ti-64 cast-and-extruded alloy. That is, any strengthening from the TiB-phase load sharing in the B-modified alloys may have been balanced by the stronger α -phase texture in the cast-and-extruded Ti-64 alloy.

The *in-situ* tensile experiment at 455°C (Figure 4.17) indicated that the B-modified alloys exhibited global yielding above their respective yield stresses, and local surface microcracks in the TiB whiskers were observed at stresses well below the global yield stress. In some cases, the TiB whisker cracks were observed at stresses more than 150 MPa below their global yield stress. A similar sequence of surface deformation events during tension has been noted at RT [Boehlert (2006)], which confirms that the TiB cracking did not inhibit ductility of the Ti-64-xB alloys. Crack initiation at ceramic particles or whiskers and propagation into Ti-64 matrices has also been observed elsewhere [Soboyejo (2004)].

The E value of α Ti is strongly anisotropic (Figure 5.5) [Zarkades (1970)]. The E value in the c-orientation is 145 GPa and the E value in directions perpendicular to the c-orientation is 100 GPa, see Figure 5.5. The cast-and-extruded Ti-64-xB alloys were strongly textured with the basal plane perpendicular to the extrusion axis. The E values

of these alloys tested in a direction parallel to the extrusion direction were thus increased due to this texture. For example, the E value of the cast-and-extruded Ti-64 alloy was 125 GPa, while the E value of the as-cast Ti-64 was 107 GPa. Increased B content would also be expected to increase the E values. However, the B addition also weakened the α phase texture for the cast-and-extruded alloys (as has been described early in this chapter) thereby reducing the contribution of the α phase texture on the E value increase. To quantify and predict the α -phase texture weakening by B addition is important, and this is the subject of the next paragraphs.

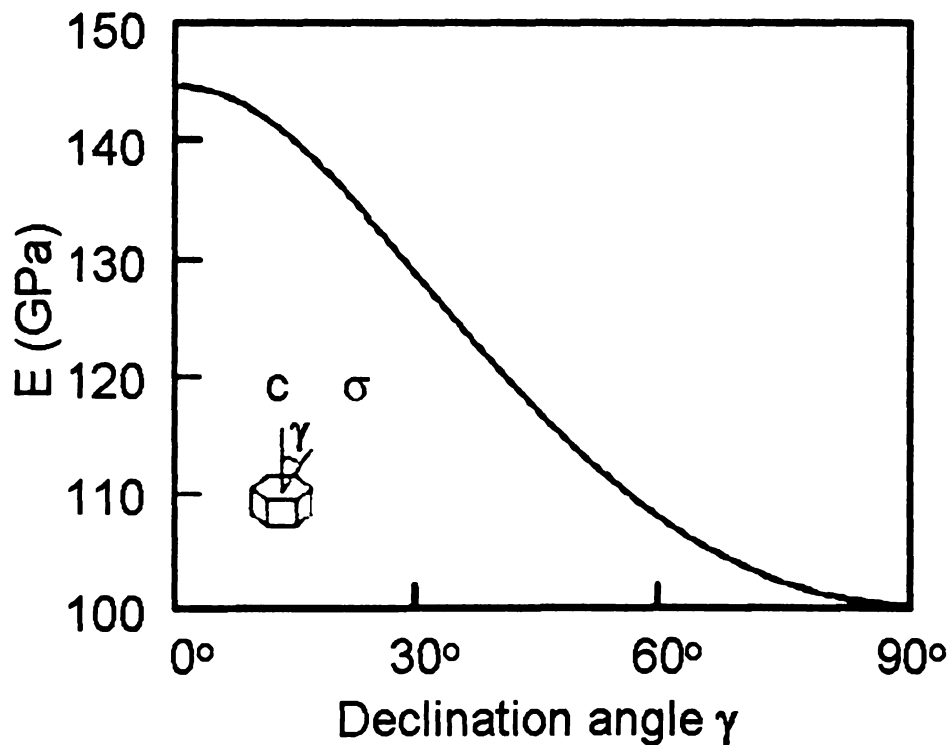


Figure 5.5. Modulus of Elasticity (E) of α -Ti single crystals as a function of declination angle. [Zarkades (1970)]

The Halpin-Tsai model [Mallick (1988)] is used here to predict the E value of the composite. The prediction of the composite E value was greater than that measured for the cast-and-extruded Ti-64-1B alloy. The decreased α -phase texture with increased B content was expected to be the cause for this discrepancy. In the Halpin-Tsai model, the longitudinal composite stiffness is expressed as:

$$E_{CL} = \frac{1 + 2(l/d)\eta_L V_f}{1 - \eta_L V_f} \cdot E_m \quad (5.1)$$

in which

$$\eta_L = \frac{E_{f,L} - E_m}{E_{f,L} + 2(l/d) E_m} \quad (5.2)$$

The E_m and E_f are the Young's modulus of the matrix and the short-fiber-reinforcement, respectively. V_f is the volume percent of the reinforcement. The l/d is the aspect ratio of the reinforcement. The subscript C denotes the composite, and subscript L denotes the longitudinal direction. In this study, the short-fiber-reinforcement is the TiB whiskers. The RT E value of TiB in the longitudinal direction was estimated to be 450 GPa [Gorsse (2003)]. The aspect ratio (l/d) of TiB whiskers in the cast-and-extruded Ti-64-1B is estimated to be 10.

Since the E value of the Ti-64 alloy is not related to grain size or α phase morphology, E value of the as-cast Ti-64 alloy (107 GPa, in Table 4.9) will be used as E_m in the Halpin-Tsai equations. Using this model, the predicted E value of the Ti-64-1B composite is 142 GPa, assuming the $E_m=125$ GPa as measured in the cast-and-extruded Ti-64 alloy. This value is larger than the measured RT E value of the cast-and-extruded

Ti-64-1B alloy (128 GPa). The discrepancy between the estimated and measured E values is expected to be due to the less degree of texture in the α -phase in the cast-and-extruded Ti-64-1B alloy compared to that in the cast-and-extruded Ti-64 alloy. That is, the E_m value in the model (taken from the cast-and-extruded Ti-64 measurement) overestimated the E value of the matrix in the cast-and-extruded Ti-64-1B alloy. If we input the measured E value of the cast-and-extruded Ti-64-1B alloy as E_{CL} (i.e. $E_{CL}=128$ GPa), we can use this model to predict an E_m value of 110 GPa. This value appears to be a reasonable estimate of the E value in the matrix of the cast-and-extruded Ti-64-1B composite because it is less than the E value of cast-and-extruded Ti-64 alloy (125 GPa), yet it is greater than the E value of the as-cast Ti-64 alloy ($E=107$ GPa).

1.3 PM Processed Ti-64-1B Alloys

Although the α -phase morphology in the PM rolled (duplex) and as-cast (lenticular) Ti-64-1B alloys was different, the tensile strength of each of these alloys was approximately the same (see Figure 4.19). This result is expected since strength is virtually unaffected by the shape of the α phase [Froes (2004)]. In addition, the E values of these two alloys were quite close as well (see Table 4.10). In general, duplex microstructures exhibit higher ϵ_f values compared with fully-equiaxed microstructure [Lutjering (2007)]. This may be one reason why the PM rolled Ti-64-1B exhibited a higher ϵ_f than the as-cast Ti-64-1B alloy. The tensile properties of the PM extruded (equiaxed) Ti-64-1B alloy were similar to the cast-and-extruded (lenticular) Ti-64-1B alloy, although the α -phase morphology in these two alloys were significantly different.

Starting with the same pre-alloyed Ti-64-1B powder, the extrusion process resulted in a significantly stronger Ti-64-1B alloy than the rolling process. Similar to the cast-and-extruded Ti-64-xB alloys, the TiB phase was aligned in the extrusion direction or the PM extruded Ti-64-1B alloy. The α phase of the PM extruded Ti-64-1B alloy was also strongly textured with the basal plane perpendicular to the extrusion axis, see Figure 4.13. As the vast majority of the microstructure consisted of the α -phase, the texture of the α -phase is an important microstructural parameter for the mechanical properties. If the basal plane is either normal or parallel to the extrusion/tensile axis, then slip along the basal plane is prevented. Thus this represents a “hard orientation”. Gorsse and Miracle [Gorsse (2003)] studied the influence of TiB phase orientation on the tensile properties of PM processed Ti-64/TiB composites at RT and 300°C. Their results show that the tensile strength and E values of the composites with aligned TiB phase were always higher than composites with randomly-oriented TiB phase. Thus, in the current study, both the alignment of the TiB phase and the α -phase texture in the PM extruded Ti-64-1B alloy contributed to its higher tensile strength and E value compared with the PM rolled Ti-64-1B alloy. In addition, the smaller α grain size (equiaxed) in the PM extruded Ti-64-1B alloy also contributed to its higher strength [Lutjering (2007)]. The E value of the PM extruded Ti-64-1B alloy (117 GPa at 455°C and 142 GPa at RT) was higher than that of the cast-and-extruded Ti-64-1B alloy (113 GPa at 455°C and 128 GPa at RT). This is expected to be a result of the stronger (0002) texture in the PM extruded Ti-64-1B compared with the cast-and-extruded Ti-64-1B alloy. This is consistent with the E calculations made based on the Halpin-Tsai model. For example, considering the E_{CL} value to be 142 GPa, in Equation 5.1, an E_m value of 125 GPa is estimated. This value is

higher than the E_m value estimated for the cast-and-extruded Ti-64-1B alloy (110 GPa). This is consistent with the XRD scan (Figures 4.11f and 4.13d) on these two alloys, which indicated that the (0002) texture in the PM extruded Ti-64-1B alloy was stronger than that in the cast-and-extruded Ti-64-1B alloy.

1.4 Tensile Anisotropy in Extruded Ti-64-xB Alloys

The tensile strength in the longitudinal direction for all the extruded alloys (Ti-64-xB cast-and-extruded and Ti-64-1B PM extruded) was higher than that in the transverse direction, as has been shown in Figures 4.16 and 4.20. This tensile anisotropy resulted from the texture caused by the extrusion process, which has been described in Chapter 4. Figure 5.6 indicates that the tensile anisotropy was weakened as the B content in the Ti-64 alloy increased. At 455°C, the UTS difference between longitudinal and transverse directions decreased from 103 MPa $\rightarrow$ 87 MPa $\rightarrow$ 35 MPa as the B content increased from 0 wt.% $\rightarrow$ 0.1 wt.% $\rightarrow$ 1 wt.%, see Figure 5.5a. The UTS difference at RT followed the same trend. Similar to the trend of UTS difference, the YS at 455°C as well as at RT also decreased with the increase of B content (see Figure 5.6b). This decreasing trend of the tensile strength is reasonable [Lutjering (2007)] since the XRD data (Figure 5.1) indicated that the (0002) texture in the longitudinal direction was weakened by the increasing B content. For the cast-and-extruded Ti-64-1B alloy, the TiB phase tended to compensate for the loss of strength in the transverse direction due to the weakening texture of α phase. Thus the combined effect of both the TiB strengthening and the α -phase texture weakening resulted in the lower than expected tensile anisotropy in the cast-and-extruded Ti-64-1B alloy.

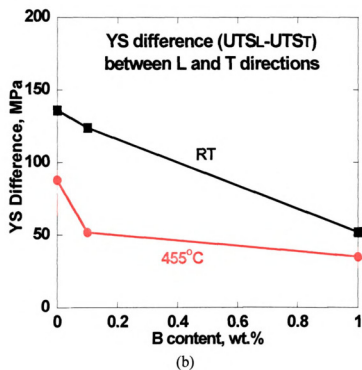
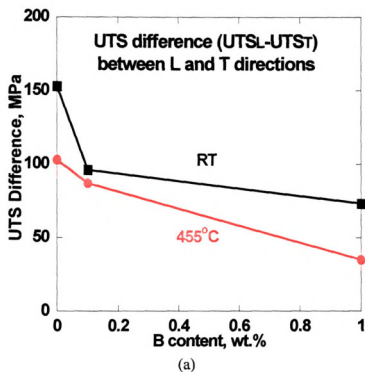
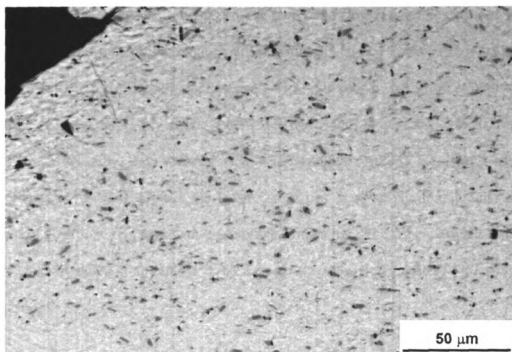
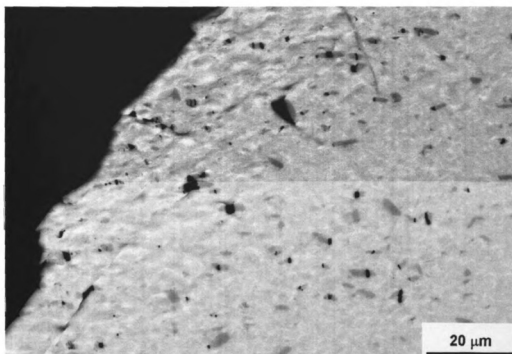


Figure 5.6. (a) UTS difference and (b) YS difference between longitudinal (L) direction and transverse (T) direction for the Ti-64-xB cast-and-extruded alloys at both RT and 455°C. The trend indicates the weakening of tensile anisotropy due to B addition.

For the PM extruded Ti-64-1B alloy, the tensile anisotropy was much stronger than that of the cast-and-extruded Ti-64-1B alloy. The UTS difference between L and T directions was 97 MPa and 167 MPa at 455°C and RT, respectively (Figure 4.20). The YS difference between L and T directions was 58 MPa and 82 MPa at 455°C and RT, respectively. The reason for this result is that the α -phase texture in the PM extruded Ti-64-1B alloy was much stronger: the (0002)/(10-10) ratio is 3.4 compared with 1.1 for the cast-and-extruded Ti-64-1B alloy. The PM rolled Ti-64-1B alloy did not exhibit tensile anisotropy (Figure 4.20). The tensile strength in the transverse direction was as high as in the longitudinal direction (also close to the tensile strength of as-cast Ti-64-1B alloy) since there was not a strong α -phase texture developed during the rolling process. The RT tensile data reported by Schwendiman [Schwendiman (2007)] on the exact same material (PM rolled Ti-64-1B alloy cut from the same rolled plate) also indicates that the longitudinal and transverse directions have comparable tensile properties. During the *in-situ* tensile tests for the extruded Ti-64-xB alloys in the transverse direction, debonding not observed, indicating a strong interface bonding between the TiB phase and the Ti-64 matrix (see Figure 5.7). This phenomenon is consistent with observations by Gorsse and Miracle [Gorsse (2003)].



(a)



(b)

Figure 5.7. SE SEM images of a PM extruded Ti-64-1B alloy tested in transverse direction. (a) Low magnification image, and (b) high magnification image. Images were taken close to the fracture surface. Notice that the TiB phase cracked extensively but there was no debonding between TiB and Ti-64 matrix.

In summary, the tensile strength in the transverse direction for the extruded Ti-64-xB alloys is lower than in the longitudinal extrusion direction. However, it is still much higher than the tensile strength of the as-cast Ti-64-1B and Ti -64 alloys at both 455°C and RT. (For as-cast Ti-64-1B alloy, UTS was 614 MPa [455°C] and 1045 MPa [RT]; for as-cast Ti-64 alloy, UTS was 435 MPa [455°C] and 900 MPa [RT]). The B addition and processing did not degrade the tensile properties in either the longitudinal or the transverse direction compared with the baseline Ti-64 alloy. Thus, the extruded Ti-64-xB alloys have the potential to replace Ti-64 for both elevated-temperature and RT structural applications.

2. As-cast Ti-6242S-xB Alloy System

For the tensile properties of as-cast Ti-6242S-xB alloys shown in Tables 4.11 and Figure 4.21, it is clear that B addition increased the tensile strength of the Ti-6242S alloy. Larger B contents led to higher YS and UTS at 455°C. The strengthening effect by B addition came from load sharing by TiB whiskers, which is similar to the Ti-64-xB alloy system [Tamirisakandala (2003)]. Since the B addition also reduced the as-cast grain size of the Ti-6242S alloy system (see Figure 5.8), the Hall-Petch mechanism also contributed to the strengthening. This mechanism was suggested by Sen *et al.* [Sen (2007)] in the as-cast Ti-64-xB ($x=0, 0.05, 0.1$, and 0.4) alloys.

For the as-cast Ti-6242S-xB alloy system, Ti-6242S-0.1B alloy exhibited the highest ϵ_f value (10.3%). This was due to the grain refinement effect by B addition and it improved the workability of the Ti-6242S alloy [Tamirisakandala (2006)]. However, when the B content was higher, the ϵ_f value for the as-cast Ti-6242S-xB alloys decreased. The increase of ϵ_f by grain refinement was offset by large amount of brittle TiB whiskers, which is similar to the situation in the as-cast Ti-64-xB alloy system.

Since B addition also reduced the α -colony size in the as-cast Ti-6242S alloy system (see Figure 5.8). This is also considered to have contributed to the strengthening [Lutjering (2007)]. The α -lath width of the as-cast Ti-6242S-xB alloys was quite stable and independent of B concentration, so this factor was not expected to have affected the tensile properties.

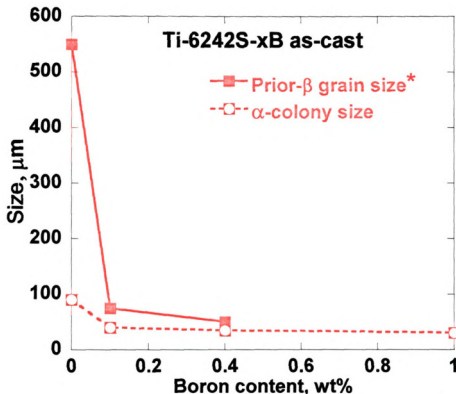


Figure 5.8. Effect of B addition on the prior-β grain size and α colony size of as-cast Ti-64 alloy. [Tamirisakandala (2005)]

The 455°C tensile strengths of the Ti-6242S-xB alloys (Table 4.11) were significantly greater than those for the Ti-64-xB system (Table 4.8) at a given B concentration. The ϵ_f values for the as-cast Ti-6242S alloy was slightly larger than that for the as-cast Ti-64 alloy, while the alloys containing 1 wt.% B exhibited similar ϵ_f values. Thus B concentrations of 0.4 wt.% or more results in ϵ_f values less than 2.5% for both Ti-6242S-xB and Ti-64-xB castings. The greatest ϵ_f values for both alloy systems were observed for 0.1 wt.% B additions. The tensile-deformation behavior in both of the Ti-6242S-xB and Ti-64-xB alloy systems was similar. The alloys exhibited global yielding above their respective yield stresses and surface microcracks in the TiB whiskers

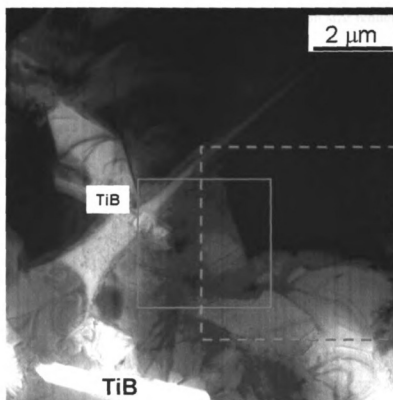
were observed at stresses well below the global yield stress. In some cases, the TiB whisker cracks were observed at stresses more than 100 MPa below their global yield stress.

D. Fatigue Behavior Discussion

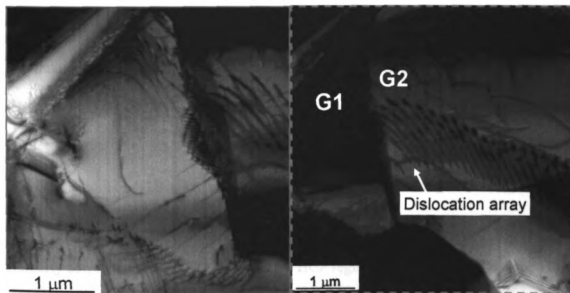
1. Ti-64-xB Alloy System

1.1 As-cast Alloys

The improved average fatigue lives of the B-modified alloys compared with Ti-64 is believed to be due to the microstructural differences. Ti-64 exhibits superior high-cycle smooth bar fatigue lives when the slip length is small [Peters (1981), Stubbington (1974), Williams (1981)]. Since a grain boundary can act as an effective obstacle to dislocation motion, reducing the as-cast grain size of Ti-64 alloy will help reduce the dislocation slip length. Figure 5.9 illustrates a larger dislocation density at the grain boundary area of a fatigue-tested as-cast Ti-64-1B alloy. Figure 5.9c illustrates a dislocation array piled up at the grain boundary. Large dislocation densities were only observed in the area close to grain boundaries. In the current study, the B additions reduced the as-cast grain size by almost an order of magnitude and a significant decrease in the α colony size was also observed. This grain refinement could have significantly enhanced the fatigue resistance. The smaller α -colony size has been shown to be beneficial to the fatigue resistance [Lutjering (2007)]. The fatigue strength of Ti-64 has been directly correlated to the grain size dependent (α grain size or α -lath width) yield stress [Wagner (2003)]. In addition, the TiB whiskers were expected to have effectively decreased the slip length (and impede dislocation motion) compared to the Ti-64 alloy and thereby increased the fatigue resistance. This phenomenon was observed in PM rolled Ti-64-1B alloy, which will be discussed later. The small change in α -lath width observed in the current study was not expected to have held as prominent of an effect on the fatigue lives since it only varied to a small degree (less than 0.7 μm).



(a)



(b)

(c)

Figure 5.9. TEM bright field image of a fatigue-tested as-cast Ti-6Al-4V alloy ($N_f=14,120$ cycles, $\sigma_{max}=450\text{MPa}$). (a) Grain boundary area (TiB phase can be seen), (b) Higher magnification image in the solid rectangle of (a), a large amount of dislocations can be seen, and (c) Higher magnification image in the dashed rectangle of (a), dislocations piled up at the grain boundary.

The 0.1B addition resulted in a significant as-cast grain size reduction from 1700 μm for Ti-64 to 200 μm for Ti-64-0.1B. It also resulted in increased ϵ_f compared to the other alloys studied (see Tables 4.8 and 4.9). For maximum stresses greater than 400 MPa, most of the fatigue lives presented here were considered to be in the low-cycle-fatigue (LCF) regime ($<100,000$ cycles) where greater ϵ_f and damage tolerance tends to correspond to greater fatigue lives [Hertzberg (1996)]. Thus the significantly greater ϵ_f value of the Ti-64-0.1B alloy (11.8% at 455°C) compared with that for Ti-64-1B (2.5% at 455°C) is expected to have enhanced its fatigue life. This may be related to the longer average fatigue lives of the Ti-64-0.1B alloy than those of the Ti-64-1B alloy.

Comparing the S-N behavior of the B-containing alloys (Figure 4.26a), the Ti-64-0.1B alloy significantly outperformed the Ti-64-1B alloy at high stresses (LCF regime), while the N_f values were closer at lower applied stresses. For maximum applied stresses less than 400MPa, most of the N_f values were considered to be in the high-cycle-fatigue (HCF) regime ($>100,000$ cycles) where increased tensile strength tends to correspond to greater fatigue lives and greater fatigue endurance limits or thresholds [Hertzberg (1996)]. The Ti-64-1B alloy exhibited the greatest tensile strength and the lowest ϵ_f value at 455°C (see Table 4.8). Thus the increased strength of the Ti-64-1B alloy is similarly correlated with the N_f values in the HCF regime being closer to those for Ti-64-0.1B. The load sharing by the TiB phase was expected to be responsible for the greater average fatigue lives of Ti-64-1B compared with those for Ti-64. Since the strength (8.0 GPa [Boehlert (2009)] and E (470~485 GPa [Gorsse (2003)]) of the TiB phase are significantly higher than those for the α and β phases, much of the load placed on the

alloy was supported by the TiB phase. As a consequence of the load-sharing by the TiB phase, the stress in the α and β phases is lower. The highly-loaded TiB phase can break at locations where stresses are large and where flaws developed. Load sharing was also explained to be the reason for the increased creep resistance of B-modified Ti-64 cast alloys compared with Ti-64 castings, which will be discussed later in this chapter.

It is also noted that the fatigue fracture surface of the Ti-64-0.1B as-cast alloy was much rougher than that of the Ti-64-1B as-cast alloy, which implies a more tortuous and longer crack front in the Ti-64-0.1B as-cast alloy. Hence the crack driving force per unit length of the crack front will be smaller than for the flatter crack front in the Ti-64-1B as-cast alloy. This may have contributed to the enhanced fatigue resistance of the Ti-64-0.1B as-cast alloy since the crack growth resistance of a material with a tortuous crack front will be larger than that for a material with a flat crack front [Schijve (2009)].

1.2 Cast-and-extruded Alloys

Comparing Figure 5.2 and 5.3, the grain size and a colony size for the cast-and-extruded alloys are much finer than for the as-cast alloys. Figure 5.4 also indicates that the α lath was four times finer than the as cast alloys. These microstructural features are expected to cause significantly improved fatigue resistance of the cast-and-extruded Ti-64-xB alloys.

The α -phase texture of the cast-and-extruded alloys was expected to be the primary reasons for the increased fatigue strength of the cast-and-extruded alloys compared to the as-cast alloys. The texture of the anisotropic α -phase is critical to its mechanical behavior. At elevated temperatures, the α phase deforms due to 'a' and some

'c+a' slip, where 'a' slip is always dominant for α -Ti [Wagner (2003)]. Paton *et al.* [Paton (1976)] investigated the temperature dependence of the critical resolved shear stress (CRSS) for slip with 'a' and 'c+a' Burgers vectors in single crystals of Ti-6.6Al, see Figure 5.10. It was shown that at 455°C, the CRSS for 'c+a' slip is approximately 400 MPa, which is higher than the 100 MPa for 'a' slip [Paton (1976)]. When the basal plane is perpendicular to the tensile axis, as was the case for the textured cast-and-extruded alloys, the 'a' slip is prevented. Thus fatigue deformation is only possible in 'c+a' slip for this case. This is expected to be a major reason for the increased fatigue resistance of the cast-and-extruded alloys compared to the as-cast alloys, which were not as strongly textured. The experimental observations indicated that for all the cast-and-extruded alloys, the Ti-64 alloy exhibited the highest preference for the α -phase to be oriented at 90° with respect to the extrusion axis. This is believed to be the reason for its enhanced fatigue resistance compared with the B-containing cast-and-extruded alloys. Thus variant selection may have been influenced by the B content and caused different (0002) texture strength of α -phase. Although it has been suggested that the α -colony size plays an important role in the fatigue crack growth behavior [Lutjering (2007)], it was difficult to isolate the effect of the decreasing α -colony size (which decreased with increased B content) on the fatigue lives for the cast-and-extruded alloys.

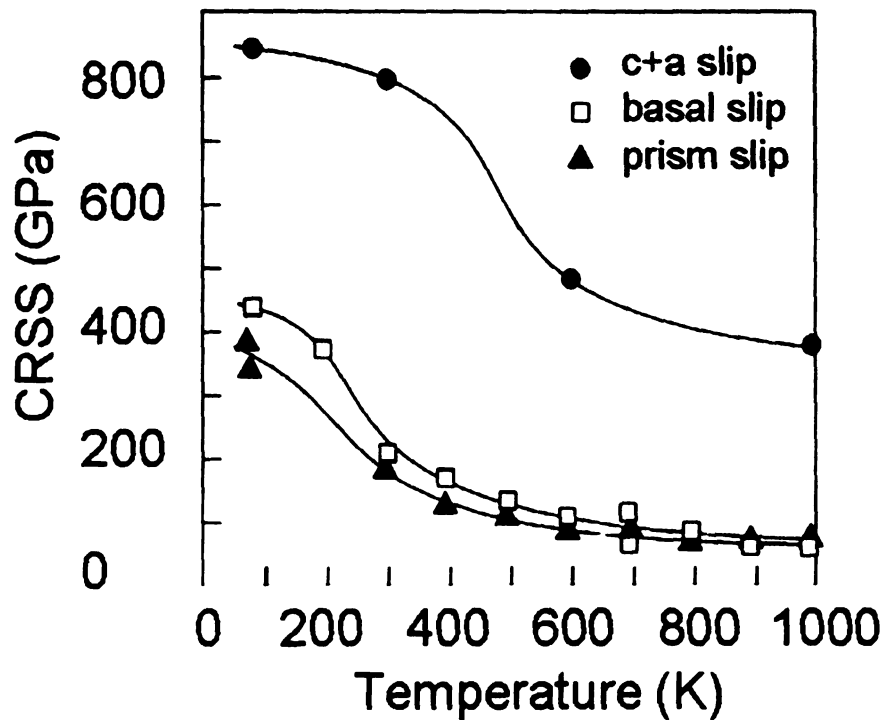


Figure 5.10. Temperature dependence of CRSS for slip with 'a' and 'c+a' vectors in single crystals of Ti-6.6Al. [Paton (1976)]

1.3 PM Processed Ti-64-1B Alloys

As has been described previously in Chapter 4, the PM processed Ti-64-1B alloys exhibited better fatigue resistance than the as-cast and cast-and-extruded Ti-64-1B alloys (Figure 4.39). The PM extruded alloy exhibited extraordinary fatigue performance compared with all other alloys, but its tensile strength is about the same as the cast-and-extruded alloy. Although the tensile strength of the PM rolled Ti-64-1B alloy is much lower than that of the cast-and-extruded Ti-64-1B alloy, its average fatigue lives was still higher than that of the cast-and-extruded alloy. In general, there are several microstructural features that improve the fatigue resistance of Ti alloys. First and foremost, PM processing induced an equiaxed α grain and this resulted in enhanced

fatigue lives compared to cast metallurgy. It is well established that low-cycle fatigue (LCF) behavior is favored by equiaxed α morphology [Froes (2004)] and smaller grain size (equiaxed α) results in better fatigue properties [Lutjering (2007)]. The equiaxed α grain size in the PM extruded Ti-64-1B alloy (3.6 μm) is much smaller than that in the PM rolled Ti-64-1B alloy (7.4 μm). This is one reason why the fatigue resistance of PM extruded alloy was superior to the PM rolled alloy. It has been reported that reducing the equiaxed α grain size in Ti-64 alloy from 12 to 2 μm increased fatigue strength from 560 to 720 MPa [Peters (1983), Trojahn (1980)]. Secondly, the α -texture (basal plane perpendicular to tensile axis) also enhanced the fatigue resistance in the PM extruded Ti-64-1B alloy, which is similar to the situation in cast-and-extruded Ti-64-xB alloys. As has been described earlier in this chapter, the (0002) texture of the α phase in the PM extruded Ti-64-1B was stronger than that in the cast-and-extruded Ti-64-1B alloy. This stronger texture leads to less dislocation activity and thus enhanced the fatigue resistance of the PM extruded Ti-64-1B compared to the cast-and-extruded Ti-64-1B alloy. In addition, the TiB whiskers decreased the slip length (by impeding dislocation motion). Figure 5.11 illustrates that large amount of dislocations were trapped between two TiB whiskers in the fatigue tested PM rolled Ti-64-1B alloy.

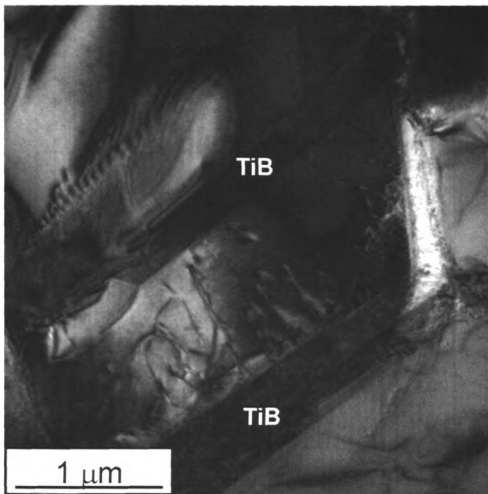


Figure 5.11. TEM bright field image of a fatigue-tested PM rolled Ti-64-1B alloy ($N_f=12,998$ cycles, $\sigma_{max}=600$ MPa). Large amount of dislocations were trapped between two TiB whiskers.

Hertzberg [Hertzberg (1996)] summarized the processing effect on the RT fatigue properties of Ti-64 alloy, see Figure 5.12. The wrought material exhibited the greatest fatigue strength, followed by HIPped pre-alloyed powder and cast alloys. From this plot, it is expected that the wrought Ti-64 alloy using pre-alloyed powder would have even better fatigue properties than the wrought alloy. The following order ranks the processing effects on the fatigue lives of the Ti-64-1B alloy at 455°C from greatest

fatigue strength to weakest fatigue strength: PM extruded > PM rolled > cast-and-extruded > as-cast. This follows a similar trend to that for the Ti-64 alloy at RT.

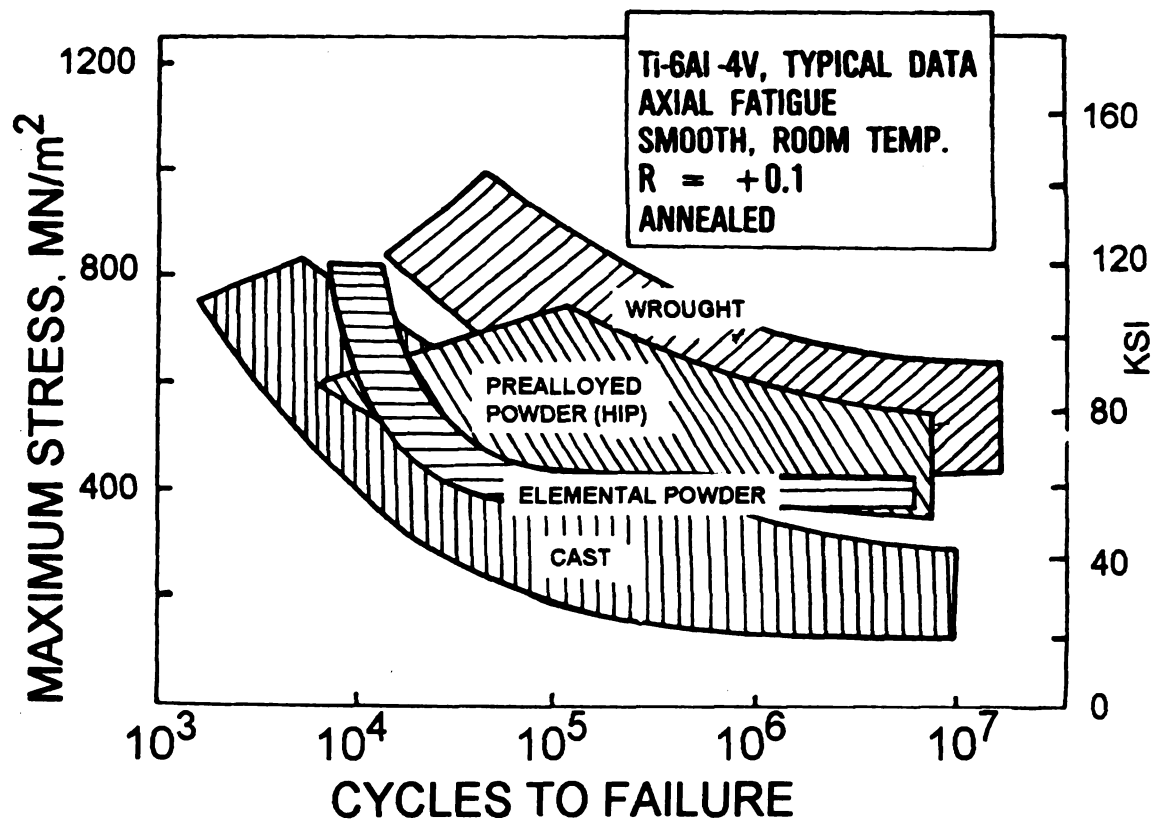


Figure 5.12. Typical RT fatigue data for Ti-64 alloy with different processing [Hertzberg (1996)]

2. As-cast Ti-6242S-xB Alloy System

Small B additions resulted in a significant as-cast grain size reduction from 550 μm for Ti-6242S to 75 μm for Ti-6242S-0.1B. It also resulted in an increase in the ϵ_f value (see Table 4.11). For maximum applied fatigue stresses greater than 400 MPa, most of the fatigue lives presented here were considered to be in the low-cycle-fatigue regime ($<100,000$ cycles) where the greater the alloy's ϵ_f and damage tolerance tends to correspond to greater fatigue lives [Sansoz (2003)]. Thus the significantly greater ϵ_f value of the Ti-6242S-0.1B alloy compared with that for Ti-6242S-1B is expected to have enhanced its fatigue life. This may explain why the average fatigue lives of Ti-6242S-0.1B were longer than those for Ti-6242S-0.4B and Ti-6242S-1B. In particular, the poor fatigue crack growth resistance of the Ti-6242S-1B alloy was noteworthy.

The higher average fatigue lives of the Ti-6242S-0.1B alloy compared with Ti-6242S is probably due to the combination of the significantly increased tensile strength and ϵ_f values exhibited by the Ti-6242S-0.1B alloy. The B effect on fatigue lives for the Ti-6242S alloy system is similar to that for the Ti-64 alloy system. In the as-cast Ti-6242S-xB alloy system, B additions also reduced the as-cast grain size and a colony size significantly. This grain refinement is expected to have contributed to both the increased tensile and fatigue strength, which is similar to the situation in the as-cast Ti-64-xB alloys. In addition, the TiB whiskers were expected to have effectively decreased the slip length (and impede dislocation motion) compared to the Ti-6242S alloy and thereby increased the fatigue resistance. The α -lath width might also play a role in the fatigue lives. For the B-containing alloys, the 0.1B alloy exhibited the finest α -lath width size and this

alloy exhibited the greatest average fatigue lives, while the 1B alloy exhibited the greatest α -lath width and the lowest average fatigue lives. It is noted that McBagonluri *et al.* [McBagonluri (2005)] studied the fatigue crack growth behavior for equiaxed and lenticular Ti-6242S microstructures and they found that the lenticular microstructure, which had significantly finer α -lath widths, exhibited significantly lower fatigue crack growth rates. Although it has been suggested that the α -colony size plays an important role in the fatigue crack growth behavior [Lutjering (2007)], it was difficult to isolate the effect of the decreasing α -colony size (which decreased with increased B content) on the fatigue lives in the as-cast Ti-6242S-xB alloy system as increased B contents resulted in lower ε_f values which could have overshadowed the effects of α -colony sizes on the fatigue lives.

3. Fatigue Endurance Limits of the Studied Alloys

The 455°C fatigue endurance limits (EL) of all studied alloys are listed in Table 5.1. For Ti alloys, the typical EL/UTS ratio lies between 0.5-0.7 [Hertzberg (1996)]. Processing and B-addition significantly changed the EL/UTS ratio of the Ti-64 and Ti-6242S alloy systems. It is noted that only the ratio of the PM extruded Ti-64-1B alloy (0.75) in this table exceeded 0.7. For the other B-modified alloys, the EL/UTS ration was typically maintained between 0.5-0.7.

Table 5.1. The 455°C Fatigue Endurance Limits of the Studied Alloys

Alloy	Processing	EL, MPa	UTS, MPa	EL/UTS
Ti-64	as-cast	<250	435	<0.57
Ti-64-0.1B	as-cast	350	552	0.63
Ti-64-1B	as-cast	300	614	0.49
Ti-64	cast-and-extruded	400	750	0.53
Ti-64-0.1B	cast-and-extruded	<450	747	<0.60
Ti-64-1B	cast-and-extruded	450	785	0.57
Ti-64-1B	PM rolled	450	702	0.64
Ti-64-1B	PM extruded	600	804	0.75
Ti-6242	as-cast	<350	569	<0.62
Ti-6242-0.1B	as-cast	450	660	0.68
Ti-6242-0.4B	as-cast	400	667	0.60
Ti-6242-1B	as-cast	<350	707	<0.50

E. Creep Behavior Discussion

1. Ti-64-xB Alloy System

1.1 As-cast and Cast-and-extruded Alloys

1.1.1 Creep Behavior

Since the stiffness and strength of the TiB whiskers are significantly higher than that of the α and β phases, much of the load placed on the alloy is supported by the TiB whiskers. Thus, the creep resistance of the Ti-64 alloy is expected to increase with an increase in B content for a given $\alpha+\beta$ microstructure. During the loading phase of the creep test, load is gradually transferred from the α and β phases to the TiB whiskers. As a consequence of load-sharing by the TiB whiskers, stress relaxation in the α and β phases occurs. The highly-loaded whiskers break at locations where stresses are maximum and where flaws accumulated. At these locations, load is transferred from the TiB whiskers to the surrounding phases, as shown in the *in-situ* creep observation in Chapter 4. The TiB whiskers were also expected to impede dislocation motion through the continuous $\alpha+\beta$ microstructure, and thereby increase the creep resistance. Several studies on the creep behavior of Ti-B alloys also found that TiB whiskers act as strong barriers to dislocation movement and sites of dislocation pileup [Rhee (2003), Kawabata (2004), Kumari(2004)]. The impediment of dislocations motion by TiB phase was also found in a fatigue tested PM rolled Ti-64-1B alloy (Figure 5.11), in which a large amount of dislocations were trapped between two TiB whiskers. However, the $\alpha+\beta$ microstructure was expected to have played a significant role in the creep resistance, as it

consisted of at least 94 vol.% of the microstructure. In particular, the α -phase alone consists of up to 80% of the microstructure.

The texture of the anisotropic α -phase is critical to its mechanical behavior. At elevated temperatures, the α phase deforms due to 'a' and some 'c+a' slip, where 'a' slip has shown to be dominant for Ti_3Al alloy microstructures [Mishra and Banerjee (1990)]. When the basal plane is perpendicular to the tensile axis, as was the case for the cast-and-extruded alloys, the 'a' slip is prevented. Thus creep deformation, due to dislocation climb, is only possible for 'c+a' slip in this case. This is expected to be another reason for the lower creep rates and increased creep resistance of the cast-and-extruded alloys compared to the as-cast alloys, which were not strongly textured. The experimental observations indicated that for all the cast-and-extruded alloys, the Ti-64 alloy exhibited the highest preference for the (0002) of α -phase to be oriented perpendicular to the extrusion axis. This is believed to be the reason for its exceptional creep resistance compared with the B-containing cast-and-extruded microstructures. Thus the α -phase variant selection may have been influenced by B content.

As the lath widths were four times smaller for the cast-and-extruded alloys compared to the as-cast alloys, the lath width is expected to have also significantly influenced the creep strains and strain rates. It is well established that in Ti-64, wider α lath (plate) leads to larger distance between obstacles for dislocation motion and lower strain hardening [Lutjering (2007)]. For TiAl alloy microstructures, decreased lath widths have resulted in significantly reduced primary creep strains and minimum strain rates [Boehlert (2002), Parthasarathy (1998), Chatterjee (2002), Chen (1999)]. Finer α lath/platelet thicknesses have also been shown to inhibit plastic flow and increase flow

stresses for the Ti-64 alloy at elevated temperatures [Semiatin (2001)]. During dislocation creep, the finely spaced lath boundaries impede dislocation motion. If the Weertman model [Weertman (1968)] of the climb of the leading dislocations at the head of the pileup constitutes the rate controlling mechanism, the lath spacing with regard to the mean free-slip path would be important to the creep rate where finer lath spacing decrease creep rates. Each of the cast-and-extruded alloys exhibited similar lath widths. Thus the improved creep resistance with increased B concentration for the Ti-64-0.1B and Ti-64-1B cast-and-extruded alloys could be attributed to the increase in the volume percent of the TiB whiskers through load sharing as described previously.

1.1.2 Deformation Evolution

The subsurface observations of tested samples agreed with the surface deformation evolution observations. The sequence of deformation during tensile-creep deformation was similar to that found previously for tensile experiments at RT [Boehlert (2006)] and elevated-temperatures (see tensile behavior part in Chapter 4). TiB whiskers exhibited microcracks at low displacement levels. These cracks grew into macrocracks and multiplied with increased creep strain and time, see Figures 4.61 and 4.63. Localized slip in the $\alpha+\beta$ microstructure initiated after the TiB whisker cracking became extensive. Deformation continued via large strain plasticity in the $\alpha+\beta$ microstructure followed by void formation and coalescence and crack formation in the $\alpha+\beta$ phases before final ductile fracture. Cracking was particularly evident at the α/β interfaces, which has been found previously for Ti-64 alloys [Eylon (1980)]. In spite of the early microcracking of

TiB on the sample surface, lower creep strains and strain rates were observed for the alloys containing greater TiB volume percents.

1.1.3 Creep Mechanism

For stresses greater than 400 MPa and temperatures greater than 400°C, the n values were greater than or equal to 8 for the as-cast alloys. Such creep exponents are not unusual for particle-strengthened alloys. It is noted that the applied creep stresses approached or exceeded the tensile yield stress of the as-cast alloys (see Table 4.8). For the cast-then-extruded alloys, the n values were greater than or equal to 4.8 at 400°C. Such values are expected to be representative of a dislocation climb mechanism. Dislocation climb creep has been confirmed in Ti-64 alloys in creep stress and temperature regimes similar to those examined in the current study, where the n and Q_{app} values were similar to those reported here [Reis (2005), Barboza (2006)]. In the current study, the Q_{app} values ranged between 275-395 kJ/mol at $\sigma=450$ MPa for the as-cast alloys (see Figure 4.56a), and the Q_{app} values ranged between 287-301 kJ/mol at $\sigma=600$ MPa for the cast-then-extruded alloys (see Figure 4.56b). Such Q_{app} values are similar to those of lattice self-diffusion for titanium aluminides [Rusing (1996)] and are twice that for grain boundary diffusional creep in pure Ti [Malakondaiah (1981)]. Thus, dislocation climb with lattice self diffusion is expected to have been the dominant creep mechanism for all the creep conditions and alloys examined.

The environmentally-assisted cracking, which detrimentally affected the creep resistance and resulted in edge cracking (see Figure 4.60) was found to be independent of B content. Such cracking could be prevented by testing in an inert environment, such as

nitrogen, which has been shown to result in improved creep properties compared to an air environment [Clauer (1969)]. Environmental susceptibility to cracking is one of the main reasons for the limited use of the Ti-64 alloy for elevated-temperature ($T > 350^{\circ}\text{C}$) structural applications.

1.2 PM Processed Ti-64-1B Alloys

Figure 5.13 summarizes the creep strain vs. time plots of all studied Ti-64-1B alloys at (a) $455^{\circ}\text{C}/400\text{MPa}$, and (b) $455^{\circ}\text{C}/450\text{MPa}$. Although the PM extruded Ti-64-1B alloy exhibited the greatest fatigue resistance among all Ti-64-1B alloys, it exhibited the poorest creep resistance. Only the PM extruded Ti-64-1B alloy entered the tertiary creep regime within 300 hrs and it exhibited the highest steady state creep rate at $455^{\circ}\text{C}/400\text{MPa}$, see Table 5.2. Its steady state creep rate at $455^{\circ}\text{C}/450\text{MPa}$ ($5.84 \times 10^{-8} \text{s}^{-1}$) was almost as high as the as-cast Ti-64-1B, which exhibited the highest strain rate ($5.89 \times 10^{-8} \text{s}^{-1}$). The creep resistance of the PM rolled Ti-64-1B alloy was not as good as the cast-and-extruded Ti-64-1B alloy. The cast-and-extruded Ti-64-1B alloy always exhibited the lowest steady state creep rate except at $455^{\circ}\text{C}/400\text{MPa}$, see Tables 5.2 and 5.3. The difference of creep resistance resulted from different microstructure caused by different processing. The creep performance of Ti-64 alloy is favored by lenticular α phase [Froes (2004)]. In addition, Lutjering [Lutjering (2007)] summarized the grain size effect on creep strength in a fully-equiaxed Ti-64 alloy: creep strength decreases with decreasing α grain size, see Figure 5.14. Thus the poor creep resistance of the PM extruded Ti-64-1B alloy is not unexpected, although its α -phase texture should be beneficial to the creep resistance.

Based on the n values and Q values for the PM rolled Ti-64-1B alloy, the creep mechanism was suggested to be dislocation creep with lattice self diffusion.

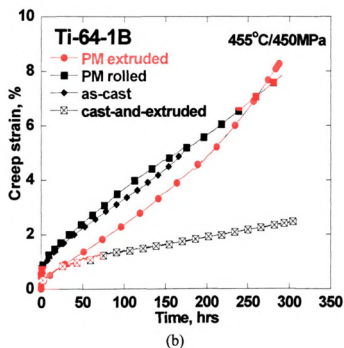
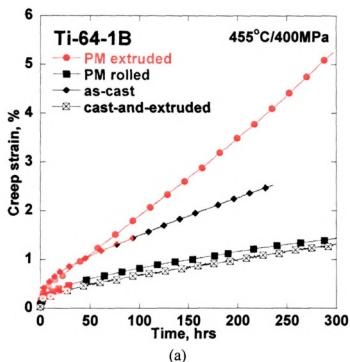


Figure 5.13. Creep strain versus time curves of the Ti-64-1B alloy of different processing. (a) At $T=455^{\circ}\text{C}$, $\sigma=400\text{ MPa}$, and (b) at $T=455^{\circ}\text{C}$, $\sigma=450\text{ MPa}$.

Table 5.2. Processing Effect on Minimum Creep Rate of Ti-64-1B Alloys at T=455°C

Creep Stress Processing	400 MPa	450 MPa
PM extruded	$4.36 \times 10^{-8} \text{ s}^{-1}$	$5.84 \times 10^{-8} \text{ s}^{-1}$
PM rolled	$6.91 \times 10^{-9} \text{ s}^{-1}$	$1.51 \times 10^{-8} \text{ s}^{-1}$
as-cast	$2.28 \times 10^{-8} \text{ s}^{-1}$	$5.89 \times 10^{-8} \text{ s}^{-1}$
cast-and-extruded	$8.87 \times 10^{-9} \text{ s}^{-1}$	$1.39 \times 10^{-8} \text{ s}^{-1}$

Table 5.3. Processing Effect on Minimum Creep Rate of Ti-64-1B Alloys at T=400°C

Creep Stress Processing	450 MPa	500 MPa	550 MPa	600 MPa
PM extruded	$1.11 \times 10^{-9} \text{ s}^{-1}$	N/A	N/A	N/A
PM rolled	$1.12 \times 10^{-9} \text{ s}^{-1}$	$3.33 \times 10^{-9} \text{ s}^{-1}$	$5.40 \times 10^{-9} \text{ s}^{-1}$	$7.81 \times 10^{-9} \text{ s}^{-1}$
as-cast	$1.43 \times 10^{-9} \text{ s}^{-1}$	$2.14 \times 10^{-9} \text{ s}^{-1}$	$6.35 \times 10^{-9} \text{ s}^{-1}$	$1.63 \times 10^{-8} \text{ s}^{-1}$
cast-and-extruded	$1.04 \times 10^{-9} \text{ s}^{-1}$	$1.33 \times 10^{-9} \text{ s}^{-1}$	$2.20 \times 10^{-9} \text{ s}^{-1}$	$4.09 \times 10^{-9} \text{ s}^{-1}$

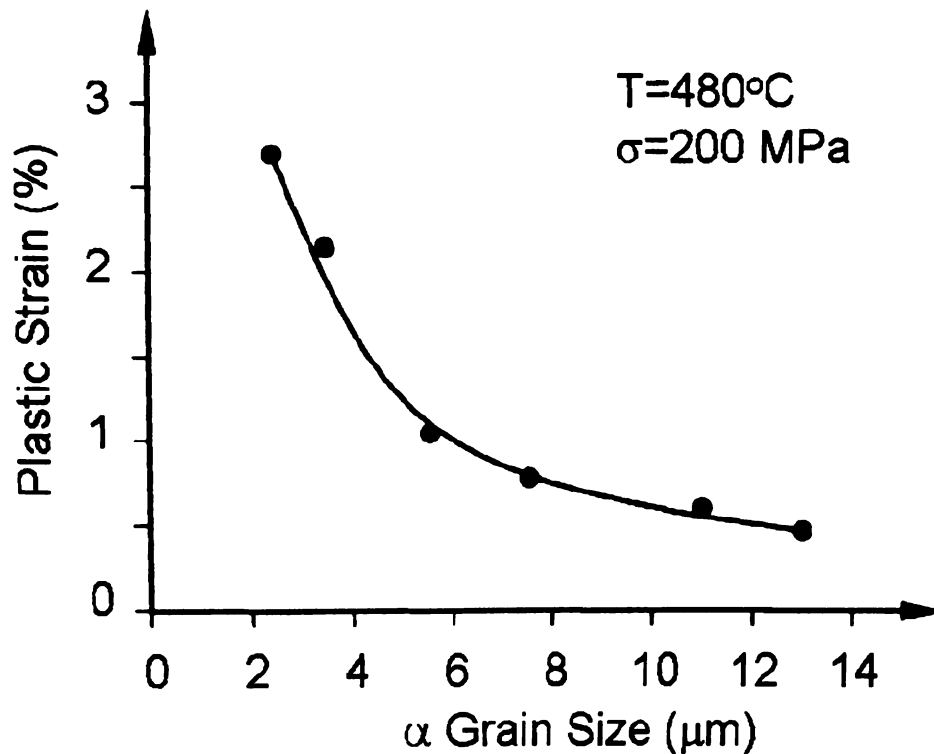


Figure 5.14. Creep strain (labeled as Plastic Strain in the figure) after 100 hrs as a function of α grain size of fully equiaxed microstructures of Ti-64 (final aging treatment: 24 hrs at 500°C). [Lutjering (2007)]

2. As-cast Ti-6242S-xB Alloy System

The Ti-6242S alloy system was developed after the Ti-64 alloy system with the intention of being a higher-temperature alloy capable of being used in service up to 565°C [Boyer (1994)]. Thus, the Ti-6242S alloy is inherently more creep resistant than Ti-64 and it maintains its strength at elevated temperature to a greater degree than Ti-64 due mainly to the solid-solution element additives. The minimum creep rates measured for the as-cast Ti-6242S-xB system were significantly lower than those measured for the as-cast Ti-64-xB system (see Table 5.4). This result was expected based on the superior elevated-temperature creep and tensile strength of Ti-6242S compared with that for Ti-64. However, the results of this work indicate that the TiB phase did not have as dramatic of an effect on the minimum creep rates of the Ti-6242S-xB alloy system as it had on the Ti-64-xB system. Thus, it is possible that the creep resistance of the TiB phase more closely resembles that of the Ti-6242S than the Ti-64 alloy. This along with the lower TiB phase volume fraction in the Ti-6242S-xB alloy system can explain the relatively small effect that B has on the creep resistance of Ti-6242S compared to that on Ti-64. However, no current tensile creep data is available for the TiB whiskers to confirm this.

Table 5.4. Minimum Creep Rate of Ti-64-xB and Ti-6242S-xB Alloy Systems at T=455°C/ σ =450MPa

Wt.% B	0B	0.1B	0.4B	1B
Alloy system				
Ti-64-xB	$1.24 \times 10^{-6} \text{ s}^{-1}$	$4.07 \times 10^{-7} \text{ s}^{-1}$	N/A	$5.89 \times 10^{-8} \text{ s}^{-1}$
Ti-6242S-xB	$3.30 \times 10^{-10} \text{ s}^{-1}$	$5.11 \times 10^{-10} \text{ s}^{-1}$	$4.13 \times 10^{-10} \text{ s}^{-1}$	$2.24 \times 10^{-10} \text{ s}^{-1}$

N/A: not available

The elevated-temperature tensile-deformation behavior in both of the Ti-6242S-xB and Ti-64-xB alloy systems was similar. The alloys exhibited global yielding above their respective yield stresses and surface microcracks in the TiB whiskers were observed at stresses well below the global yield stress [Boehlert (2006), Sen (2007)]. In some cases, the TiB whisker cracks were observed at stresses more than 100 MPa below the global yield stress. Slip was apparent in the α and β phases after TiB cracking had occurred. A similar sequence of surface deformation events has been noted for a B-modified Ti-64 alloy containing 1 wt.% B during room-temperature and 480°C tensile deformation [Boehlert (2006)]. Crack initiation at ceramic particles or whiskers and propagation into Ti-64 matrices has also been observed elsewhere [Soboyejo (2004), Vreeling (2002), Kloosterman (1998)]. TiB whisker cracking also occurs in B-modified Ti-64 alloys during elevated-temperature fatigue. In terms of the creep behavior, during the loading phase of the creep test, the load was transferred from the α and β phases to the TiB whiskers. As a consequence of the load-sharing by the strong and stiff TiB whiskers, stress relaxation in the α and β phases occurred. The highly-loaded whiskers broke at locations where the stresses were maximum and where flaws accumulated. At these locations, load was transferred from the TiB whiskers to the surrounding phases. This is an indication that the load was primarily placed on the TiB-phase. Once a sufficient extent of TiB cracking had occurred, the load was applied to the α and β phases allowing further deformation to occur via α - and β -phase slip. However, the load sharing ability of the TiB phase in the Ti-6242S-xB system was certainly not as significant as that for the Ti-64-xB system (see creep rates in Table 5.3). Thus the overall creep

improvement of the Ti-6242S-1B alloy compared to the Ti-6242S alloy was not as dramatic as that for the Ti-64-1B alloy compared to Ti-64.

The minimum creep rates for the Ti-6242S-1B alloy tended to be intermediate to those of the Ti-6242S-xNi system [Hayes (2002)] (see Figure 5.15). Thus small additions of B and Ni resulted in similar minimum creep rates, though the low-Ni containing alloy exhibited the lowest minimum creep rates. The n values suggested that dislocation creep was dominant for the B-modified Ti-6242S system at the studied temperature/stress levels, which is also in consistent with the result by Rhee *et al.* [Rhee (2003)].

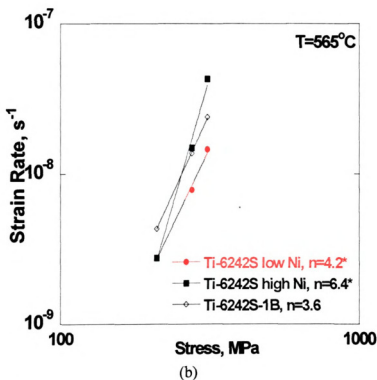
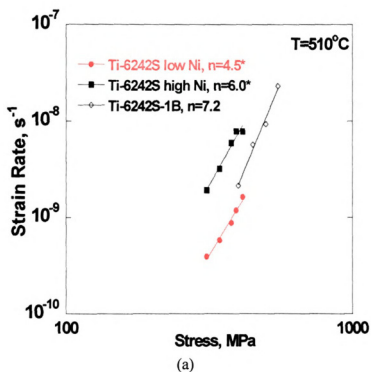


Figure 5.15. Log $\dot{\epsilon}_{\min}$ versus log σ plot at (a) $T=510^{\circ}\text{C}$ and (b) $T=565^{\circ}\text{C}$ illustrating the variation of minimum creep rate with stress for the Ti-6242S-1.0B alloy and high- and low-nickel containing Ti-6242S alloys taken from reference. *[Hayes (2002)]

F. Creep-Fatigue Interaction

As has been discussed in Chapter 4, the shift of the hysteresis with increasing cycle number in the stress versus strain plots at the marked cycle numbers (Figures 4.27, 4.40) indicates creep occurred during the fatigue tests. In this part of the discussion, the strain due to creep was estimated from the fatigue tests. The strain was taken at the peak stress of certain cycle numbers (5×10^4 , 1×10^5 , 2×10^5 , 3×10^5 , ...) and plotted with time elapsed at those cycles, see Figure 5.16 (This sample broke at 990,554 cycles and the E value dropped from 88 GPa at 100 cycle to 76 GPa at 990,000 cycles). The curve closely resembled a typical creep curve, which has primary, secondary and tertiary stages. This confirms that creep contributed to the total strain during the fatigue test. Comparing the fatigue creep curve (red) with that of the conventional creep test (blue) at the same temperature, the creep strain is always lower than those of the conventional creep test. Although the fatigue sample only “saw” the peak stress once in a cycle, the minimum creep rate in the fatigue creep test was still $5.65 \times 10^{-8} \text{ s}^{-1}$, which is close to the minimum strain rate ($7.26 \times 10^{-8} \text{ s}^{-1}$) for the conventional creep test. The large amount of dislocations generated by the fatigue process might have facilitated the creep deformation process and increased the strain rate. It is possible that the defects induced by fatigue (such as micro-voids) also increased the creep rate since fatigue damage (TiB cracking as well as the α -phase cracking) occurred for the as-cast Ti-64-xB alloys (described in Chapter 4, Figure 4.28). In Figure 5.14, the fatigue creep curve eventually entered the tertiary regime before the final fracture of the sample. The creep rate between the last two data points (corresponding cycle number: 8×10^5 to 9×10^5) was $8.46 \times 10^{-8} \text{ s}^{-1}$.

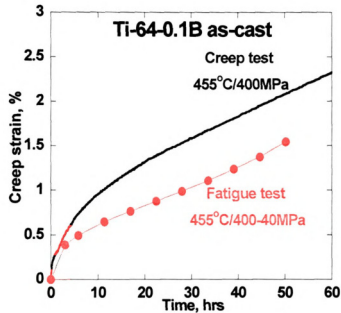


Figure 5.16. Fatigue creep curve at 455°C/400-40MPa (red) compared with the conventional creep test at 455°C/400MPa (blue) for the as-cast Ti-64-0.1B alloy. (The N_f value of the fatigue tested sample was 990,554 cycles.)

The strain due to creep was also extracted from the fatigue tests of cast-and-extruded Ti-64-xB samples, see Figure 5.17. Similar to the as-cast Ti-64-0.1B alloy, the primary and secondary creep rates in fatigue tests were both lower than those of the conventional creep test. For the cast-and-extruded Ti-64 alloy at 455°C, the minimum creep rate in the fatigue test ($N_f=840,444$ cycles) was $3.57 \times 10^{-9} \text{ s}^{-1}$, compared with $4.73 \times 10^{-9} \text{ s}^{-1}$ for the conventional creep test; for the cast-and-extruded Ti-64-0.1B alloy at 455°C, the minimum creep rate in the fatigue test ($N > 2,112,365$ cycles) was $3.38 \times 10^{-9} \text{ s}^{-1}$, compared with $5.63 \times 10^{-9} \text{ s}^{-1}$ for the conventional creep test. The fatigue creep strain was always lower in the Ti-64 alloy than in the Ti-64-0.1B alloy (red curves in Figures 5.17a and b). This phenomenon is consistent with the conventional creep test (Figure 4.54b) since the cast-and-extruded Ti-64 alloy is more creep resistant than the cast-and-extruded

Ti-64-0.1B alloy. However, the minimum fatigue creep rate in the cast-and-extruded Ti-64 alloy ($3.57 \times 10^{-9} \text{ s}^{-1}$) was slightly higher than in the cast-and-extruded Ti-64-0.1B alloy ($3.38 \times 10^{-9} \text{ s}^{-1}$) at $455^\circ\text{C}/450\text{-}45\text{MPa}$. A possible reason for this is that the fatigue damage was more severe in the cast-and-extruded Ti-64 alloy since it broke after 840,444 cycles, however the cast-and-extruded Ti-64-0.1B never broke during the fatigue test at $455^\circ\text{C}/450\text{-}45\text{MPa}$. For the cast-and-extruded Ti-64-1B alloy at 455°C , the minimum creep rate in the fatigue test ($N > 2,033,136$ cycles) was $1.94 \times 10^{-9} \text{ s}^{-1}$, compared with $8.87 \times 10^{-9} \text{ s}^{-1}$ for the conventional creep test.

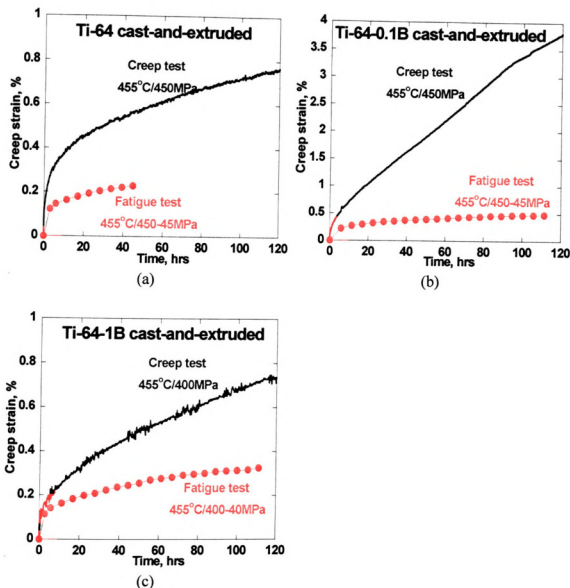


Figure 5.17. Fatigue creep curves (red) compared with conventional creep curves (blue) for the cast-and-extruded Ti-64-xB alloys at 455°C: (a) fatigue test at 450-45 MPa ($N_f=840,444$ cycles) VS. creep test at 450 MPa for Ti-64 alloy, (b) fatigue test at 450-45 MPa ($N=2,112,365$ cycles without failure.) VS. creep test at 450 MPa for Ti-64-0.1B alloy, and (c) fatigue test at 400-40 MPa ($N=2,033,136$ cycles without failure.) versus creep test at 400 MPa for Ti-64-1B alloy.

The fatigue creep curves (red) versus conventional creep curves (blue) PM rolled Ti-64-1B alloy at 455°C are plotted in Figure 5.18. In addition, the fatigue creep curves at different fatigue stress levels are shown in Figure 5.19a (Ti-64-1B PM rolled alloy) and Figure 5.19b (Ti-64-1B PM extruded alloy). Similar to that in conventional creep tests, higher stress levels resulted in higher creep rates.

In summary for the Ti-64-xB alloys, the strain due to creep in the fatigue tests at 455°C was significant. Even though the fatigue samples only experienced the highest stress once in a cycle, the fatigue creep strain rates were close to or even higher than those for the conventional creep tests at the same stress levels (equal to the maximum stress in fatigue tests). In the conventional creep tests, secondary creep starts when the dislocations substructure is stable in the samples. In the elevated-temperature fatigue tests, the large amount of dislocations generated by fatigue could facilitate the creep deformation process and it is possible that the dislocation substructure never becomes stable. Thus the creep strain rate during the fatigue tests may never enter the “steady-state”. In addition, defects induced by fatigue (such as micro-voids) could also increase the creep rate.

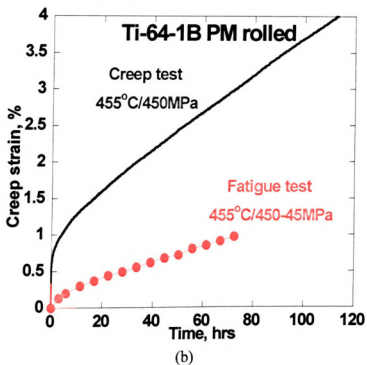
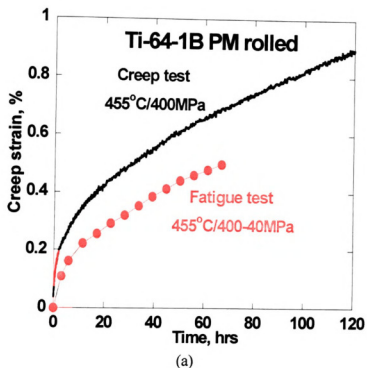
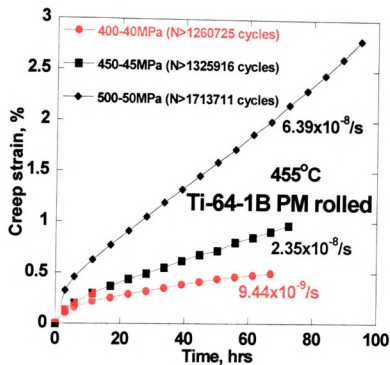
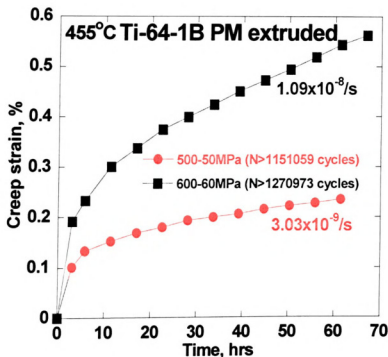


Figure 5.18. Fatigue creep curves (red) compared with conventional creep curves (blue) for the PM rolled Ti-64-1B alloy at 455°C: (a) fatigue test at 400-40 MPa ($N=1,260,725$ cycles without failure) versus creep test at 400 MPa, and (b) fatigue test at 450-45 MPa ($N=1,325,916$ cycles without failure.) VS. creep test at 450 MPa.



(a)



(b)

Figure 5.19. Fatigue creep curves at 455°C at different stress levels for (a) PM rolled Ti-64-1B alloy, and (b) PM extruded Ti-64-1B alloy. Minimum fatigue creep rates are labeled next to the corresponding curves.

CHAPTER 6

SUMMARY AND CONCLUSIONS

A. SUMMARY

The work in this dissertation constituted a systematic study on the physical and mechanical metallurgy of two B-modified $\alpha+\beta$ Ti alloy systems, namely Ti-64 and Ti-6242S. The effect of different B additions on the microstructure, creep, and elevated-temperature fatigue properties were investigated for the as-cast Ti-64-xB (x=0, 0.1 and 1 wt.%) alloys and Ti-6242S-xB (x=0, 0.1, 0.4 and 1 wt.%) alloys. In addition, the effect of processing (cast, cast-and-extruded, PM rolled, and PM extruded) on the mechanical behavior was also investigated for the Ti-64-1B alloy. Creep-fatigue interactions were also evaluated in the B-modified Ti-64 alloy system. The list of peer-reviewer journal articles that have been published regarding this work over the course of the last three years include the following:

W. Chen, C.J. Boehlert, J.Y. Howe, E.A. Payzant. The elevated-temperature mechanical behavior of as-cast and wrought Ti-6Al-4V-1B. Acta Materialia (2010), (under review).

W. Chen and C.J. Boehlert. Characterization of the microstructure, tensile and creep behavior of powder metallurgy processed and rolled Ti-6Al-4V-1B Alloy. Key Engineering Materials, Vol. 436 (2010), pp. 123-130.

W. Chen, C.J. Boehlert. The 455°C tensile and fatigue behavior of boron-modified Ti-6Al-2Sn-4Zr-2Mo-0.1Si. International Journal of Fatigue, Vol.32, Issue 5 (2010), pp. 799-807.

W. Chen, C.J. Boehlert, E.A. Payzant, J.Y. Howe. The effect of processing on the 455°C tensile and fatigue behavior of boron-modified Ti-6Al-4V. International Journal of Fatigue, Vol. 32, Issue 3 (2010), pp. 627-638.

W. Chen, C.J. Boehlert. The effect of boron on the elevated-temperature tensile and creep behavior of cast Ti-6Al-2Sn-4Zr-2Mo-0.1Si. Metallurgical Transactions A, Vol. 40, No. 7 (2009), pp. 1568-1578.

C.J. Boehlert, **W. Chen**. The elevated-temperature creep behavior of boron-modified Ti-6Al-4V alloys. Materials Transactions Vol. 50, No. 7 (2009), pp. 1690-1703.

W. Chen, C.J. Boehlert. The elevated-temperature fatigue behavior of boron-modified Ti-6Al-4V castings. Materials Science and Engineering A, Vol. 494 Issues 1-2 (2008) 132-138.

B. CONCLUSIONS

The conclusions for each aspect of this dissertation work are summarized as follows:

1. Microstructure

(1) The as-cast Ti-64 and Ti-6242S alloys exhibited a lenticular microstructure. B additions dramatically decreased the as-cast grain size and the α -colony size and also eliminated the continuous grain boundary α -phase network in these alloys. The α - and β -lath widths did not change significantly with the addition of B. The TiB-phase was not stoichiometric in the as-cast Ti-6242S-xB alloys as a result of the presence of small amounts of Mo and Zr. The diffusion of Mo and Zr is expected to be the cause for the lower volume fractions of TiB-phase whiskers in the Ti-6242S-xB alloy system compared to the Ti-64-xB alloy system. There was not any detectable amount of B in either the α or β phases and the TiB whiskers exhibited slightly more B than Ti when compared on the atomic percent basis.

(2) The cast-and-extruded Ti-64-xB alloys also exhibited a lenticular microstructure. The β -phase field extrusion aligned the TiB whiskers in the extrusion direction and significantly reduced the grain size, α -colony size and the α - and β -lath widths of the Ti-64-xB alloys. The lath widths were independent of B content and were approximately four times smaller than those for the as-cast. The significantly finer lath widths for the cast-and-extruded alloys were likely a result of the extrusion procedure followed by faster cooling than that for the as-cast melt. The large amount of dislocations produced during the extrusion process likely acted as effective sites for heterogeneous nucleation of the α -laths during cooling.

(3) The microstructure of PM processed Ti-64-1B alloys was significantly different from that of the as-cast and cast-and-extruded Ti-64-1B alloys. The PM rolled Ti-64-1B alloy exhibited a duplex microstructure, which contained both lenticular and equiaxed α phase. It is suggested that the equiaxed α phase was introduced by the rolling process and the following annealing treatment at 760°C. The α - and β -lath widths of the PM rolled Ti-64-1B alloy were intermediate to those of the as-cast Ti-64-1B alloy and the cast-and-extruded Ti-64-1B alloy. The PM extruded Ti-64-1B alloy exhibited an equiaxed microstructure and the equiaxed α grain size was significantly finer than that of the PM-rolled Ti-64-1B alloy. The α phase morphology difference in the PM rolled and PM extruded Ti-64-1B alloys were the result of different thermo-mechanical processing routes.

(4) The β -phase field extrusion is expected to have aligned the $\{110\}$ planes of β -phase perpendicular to the extrusion axis. Thus after the $\beta \rightarrow \alpha$ phase transformation (according to the Burgers orientation relationship), a strong α -phase texture was developed in the cast-and-extruded Ti-64-xB alloys and the PM extruded Ti-64-1B alloy. The EBSD and XRD data indicated that the α -phase basal plane (0002) was predominately oriented perpendicular to the extrusion axis. B additions tended to weaken this (0002) texture in the cast-and-extruded Ti-64-xB alloys. The as-cast Ti-64-xB, as-cast Ti-6242S-xB alloys, and the PM rolled Ti-64-1B alloy did not exhibit a strong α -phase texture.

2. Tensile Properties

(1) The 455°C tensile strength and Young's modulus values of the as-cast Ti-64-xB and Ti-6242S-xB alloys increased with increased B concentrations. The strengthening effect in these two alloy systems resulted from both the load sharing by the TiB phase and grain refinement effect in the $\alpha+\beta$ microstructure. The as-cast Ti-6242S-xB alloys exhibited significantly higher UTS values than the as-cast Ti-64-xB alloys at 455°C. At 455°C, 1 wt.% of B addition resulted in ϵ_f values less than 2.5% in both alloy systems. From a ductility point of view, 0.1 wt.% of B may be optimal in these two as-cast alloy systems.

(2) The 455°C YS and UTS values of the cast-and-extruded Ti-64-xB alloys were significantly higher than the as-cast Ti-64-xB alloys. This is a result of the smaller grain size, α -colony size, and α -lath width in the cast-and-extruded Ti-64-xB alloys. The α -phase texture increased the E values of the cast-and-extruded Ti-64-xB alloys. The strengths of the cast-and-extruded Ti-64-xB alloys were insensitive to B content and their ϵ_f values were all over 8% at 455°C and 6% at RT. *In-situ* tensile tests at 455°C indicates that the B-modified alloys exhibited global yielding above their yield stresses and local surface microcracks in the TiB whiskers were observed at stresses well below the global yield stress.

(3) The tensile strength of the PM rolled Ti-64-1B alloy and as-cast Ti-64-1B alloy was approximately the same since strength is virtually unaffected by α phase morphology. From a ductility point of view, duplex microstructures exhibit higher ϵ_f value compared with lenticular microstructure. This explains why PM rolled Ti-64-1B

alloy exhibited a higher ϵ_f value than the as-cast Ti-64-1B alloy. For the PM extruded Ti-64-1B alloy, the smaller equiaxed α grain size, the α phase texture and the alignment of TiB phase all contributed to its higher tensile strength and Young's modulus compared with the PM rolled Ti-64-1B alloy.

(4) The strong α -phase texture in the cast-and-extruded Ti-64-xB alloys and the PM extruded Ti-64-1B alloy resulted in the anisotropy of tensile strength. The YS and UTS for these alloys in the transverse direction were lower than in the longitudinal (extrusion) direction. Tensile anisotropy was not observed for the PM rolled Ti-64-1B alloy. As the α -phase texture became weaker with higher B content in the cast-and-extruded Ti-64-xB alloys, the tensile anisotropy became weaker. In fact, at 455°C, the UTS difference between longitudinal and transverse directions decreased from 103 MPa $\rightarrow$ 87 MPa $\rightarrow$ 35 MPa as the B content increased from 0 wt.% $\rightarrow$ 0.1 wt.% $\rightarrow$ 1 wt.%. Still, at 455°C, the UTS values of cast-and-extruded Ti-64-xB alloys in the transverse direction were at least 48% higher than the UTS of as-cast Ti-64 alloy. During the *in-situ* tensile tests for these extruded Ti-64-xB alloys in the transverse direction, debonding was not observed, indicating a strong interface bond between TiB and Ti-64 matrix.

3. Fatigue Properties

(1) The B-containing as-cast alloys exhibited longer average fatigue lives than those for as-cast Ti-64 at 455°C. This was due to decreased grain and α -colony sizes, which effectively reduced the dislocation slip length. TEM analysis on a fatigue-tested sample showed that high dislocation densities were only observed in area close to grain boundaries and dislocation pile up at a grain boundary was also observed. In addition,

TiB whiskers also effectively decreased the slip length (and impede dislocation motion). This is supported by TEM observation in which large amount of dislocations were trapped between two TiB whiskers in a fatigue tested sample. Among all as-cast Ti-64-xB alloys, the Ti-64-0.1B alloy exhibited the longest average fatigue lives. This was explained to be a result of its higher ϵ_f value and better fatigue crack growth resistance.

(2) The cast-and-extruded Ti-64-xB alloys exhibited greater fatigue resistance than the as-cast alloys. This was explained to be a result of the significantly smaller grain size, α -colony size and α -lath widths. The strong α -phase texture, within the (0002) plane predominantly perpendicular to the extrusion (tensile) axis is also an important factor for the better fatigue resistance of the cast-and-extruded Ti-64-xB alloys. In this (0002) texture, 'a' slip will be prevented since there is no resolved shear stress on the basal plane. For the cast-and-extruded alloys, Ti-64 exhibited longer average fatigue lives than B-modified Ti-64. This was explained by the greater propensity for the α -phase (0002) plane to be oriented perpendicular to the extrusion (tensile) axis for the Ti-64 cast-and-extruded alloy compared to the B-containing cast-and-extruded alloys.

(3) The PM processed Ti-64-1B alloys exhibited better fatigue resistance than the as-cast and cast-and-extruded Ti-64-1B alloys. The equiaxed α -phase introduced in the PM rolled (duplex microstructure) and PM extruded (equiaxed microstructure) alloys played a key role since LCF behavior of Ti alloys is favored by equiaxed α morphology. The PM extruded Ti-64-1B alloy exhibit much better fatigue resistance than the PM rolled Ti-64-1B alloy since the equiaxed α grain size of the former (3.6 μm) is only about half that of the latter (7.4 μm). Similar to the situation in the cast-and-extruded Ti-64-xB

alloys, the strong α phase texture in the PM extruded Ti-64-1B alloy also enhanced its fatigue resistance.

(4) Similar to the as-cast Ti-64-xB alloy system, 0.1 wt.% B addition also led to the longest average fatigue lives at 455°C for the as-cast Ti-6242S-xB alloy system. This was explained to be a result of the higher ϵ_f value and better fatigue crack growth resistance of Ti-6242S-0.1B alloy compared with the other as-cast Ti-6242S-xB alloys. The as-cast Ti-6242S-1B exhibited the lowest average fatigue lives and this was suggested to be related to the low ϵ_f value and poor fatigue crack growth resistance exhibited by the Ti-6242S-1B alloy. Compared to the as-cast Ti-64-0.1B alloy, the as-cast Ti-6242S-0.1B alloy exhibited significantly longer average fatigue lives.

(5) The *in-situ* fatigue crack growth experiment indicated that TiB could act as crack initiation sites during fatigue tests. In an *in-situ* FCG test performed on a notched Ti-6242S-0.4B alloy, cracks initially formed in the TiB whiskers and TiB/Ti-6242S interface ahead of the notch. Slip was then observed in the α -phase surrounding the cracked TiB phase followed by α -phase cracking. The crack at the TiB/Ti-6242S interface grew progressively, causing the final failure of the sample.

(6) Processing and B-addition significantly changed the EL/UTS ratio of the Ti-64 and Ti-6242S alloy systems. The PM extruded Ti-64-1B alloy exhibited the highest ratio (0.75) which exceeded the typical range of EL/UTS ratio for Ti alloys (0.5-0.7). The other B-modified alloys typically exhibited EL/UTS ratios between 0.5-0.7.

4. Creep Properties

(1) For the as-cast Ti-64-xB alloys, the beneficial aspects of the tensile strengthening translated into enhanced creep resistance with increased B content for the as-cast alloys. The as-cast Ti-64-1B alloy exhibited minimum creep strain rates that were around an order of magnitude lower than those for the as-cast Ti-64 alloy at the same applied stress and temperature. Greater B contents also increased the creep-rupture times. The enhanced creep resistance was explained to be a consequence of the load-sharing mechanism by the strong and stiff TiB whiskers that precipitate due to B addition. In addition, TiB whiskers were also expected to effectively decreased the slip length and thus impede dislocation motion during the creep deformation. Evidence of dislocations building up near TiB whiskers was found through TEM observations. The creep stress exponents (n), were greater than 8. The apparent activation energy (Q_{app}) values ranged between 275-395 kJ/mol at $\sigma=450$ MPa (see Figure 4.56a). Such values are expected to be representative of a dislocation climb with lattice self diffusion.

(2) The cast-and-extruded Ti-64-xB alloys exhibited significantly greater creep resistance than the as-cast alloys due to texture within the α phase, caused by the β -phase field extrusion, and the significantly finer lath widths. The finely spaced lath boundaries were expected to impede dislocation motions during the creep deformation. For the cast-and-extruded condition, the Ti-64 alloy exhibited greater creep resistance than the Ti-64-1B alloy, which in turn exhibited greater creep resistance than the Ti-64-0.1B alloy. This was explained by the greater propensity for the basal plan to be oriented perpendicular to the tensile (extrusion) axis for the Ti-64 cast-and-extruded alloy compared to the B-modified cast-and-extruded alloys. The improved creep resistance with increased B

concentration for the cast-and-extruded Ti-64-0.1B and Ti-64-1B alloys could be attributed to the increase in the volume percent of the TiB whiskers through load sharing. For the cast-then-extruded alloys, the n values were greater than or equal to 4.8 at 400°C. Such values are expected to be representative of a dislocation climb mechanism. The Q_{app} values ranged between 287-301 kJ/mol at $\sigma=600$ MPa, suggesting a lattice self diffusion mechanism.

(3) Among all Ti-64-1B alloys via different processing routes, the PM extruded Ti-64-1B alloy exhibited the poorest creep resistance, although its fatigue resistance was the greatest. The creep resistance of the PM rolled Ti-64-1B alloy was intermediate to the as-cast and cast-and-extruded Ti-64-1B alloys. The difference of the creep resistance was a result of different microstructure caused by different processing. The fine equiaxed α grain in the PM extruded Ti-64-1B alloy degraded its creep resistance significantly since the creep performance of Ti-64 alloy is favored by lenticular α morphology, not equiaxed α morphology. The strengthening effect by α phase texture in the PM extruded Ti-64-1B alloy was overshadowed by the equiaxed α morphology.

(4) For the as-cast Ti-6242S alloy system, the as-cast Ti-6242S-1B alloy tended to exhibit the lowest minimum creep strain rates. The creep resistance of the as-cast Ti-6242S-xB alloy system was significantly greater than that for the as-cast Ti-64-xB alloy system. However, the as-cast Ti-6242S-xB alloy system's creep resistance did not improve as significantly with increased B concentration compared to that exhibited by the Ti-64-xB alloy system. It is believed that the creep resistance of the TiB phase more closely resembles that of the Ti-6242S than the Ti-64 alloy. This along with the lower TiB phase volume fraction in the Ti-6242S-xB alloy system can explain the relatively

small effect that B has on the creep resistance of Ti-6242S compared to that on Ti-64. Based on the n and Q_{app} values at tested stress/temperature conditions, dislocation climb with lattice self diffusion was suggested to be the dominant creep mechanism.

(5) The sequence of deformation for the Ti-64-xB and Ti-6242S-xB alloys during *in-situ* tensile-creep deformation was similar to that found during the tensile experiments. TiB whiskers exhibited microcracks at low displacement levels and these cracks grew into macrocracks and multiplied with increased creep strain and time. Then localized slip in the $\alpha+\beta$ microstructure initiated after the TiB whisker cracking became extensive. Deformation continued via large strain plasticity in the $\alpha+\beta$ microstructure followed by void formation and coalescence and crack formation in the $\alpha+\beta$ phases before final ductile fracture. Cracking was particularly evident at the α/β interfaces.

5. Creep-Fatigue Interaction

(1) The strain-time evolution during the fatigue experiments resembled that of the creep experiments. Thus it was expected that creep occurred during the elevated-temperature fatigue experiments. The strain which was expected to be due to creep was extracted from the fatigue tests. This strain was always lower than that for the conventional creep tests at the same temperatures/maximum stresses combinations. Although the samples during the fatigue tests only experienced the maximum stress once in every cycle, in several cases the minimum fatigue-creep strain rate was fairly close to the minimum creep strain rate for the conventional creep test. The large amount of dislocations generated by fatigue process may have facilitated the creep deformation process and increased the strain rate. It is possible that the defects induced by fatigue

(such as micro-voids) also increased the creep rate. The fatigue-creep curve for a broken as-cast Ti-64-0.1B sample entered tertiary creep regime due to the fatigue damage occurred during the test.

C. Recommendations for Future Work

(1) The optimum B-content for the as-cast Ti-64-xB and as-cast Ti-6242S-xB alloy systems needs to be determined. Such a B-content could lead to the best combination of elevated-temperature tensile, fatigue, and creep resistance.

(2) Experiments for measuring the mechanical properties (E value, YS, and UTS) of TiB single crystals need to be designed. Currently only values from calculations are available in the literature and there is a large data scatter for the TiB E values (371~485 GPa). The experimental values are necessary for building up constitutive models to simulate the mechanical behavior of TiB-reinforced Ti alloys.

(3) In this work, TiB has been suggested to reduce the (0002) texture of α -Ti during the β -phase field extrusion. This effect needs to be confirmed and quantified.

(4) The creep-fatigue interactions in the Ti-64-xB alloy system need to be studied in greater detail. The results should be analyzed and compared with the results and theories for particle-reinforced Ti alloys in literature.

(5) The in-phase and out-of-phase thermomechanical fatigue tests need to be performed on the Ti-64-xB and Ti-6242S-xB alloy systems since materials used in aerospace applications are subjected to stresses and temperatures that can vary simultaneously.

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