Microstructural Characterization of Titanium Alloys with Fretting Damage

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This thesis is dedicated to every woman who came before me and found the courage to pursue their goals, as well as to the men who supported them. Your commitment paved the way for future generations of women engineers and scientists.
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SUMMARY

The primary focus of this work is to understand the role of microstructure in the fretting damage process and develop quantifying measures in fretting damage accumulation in a dual phase Ti-6Al-4V as well as two single phase materials: commercially pure titanium (CP-Ti), which consists of pure α-phase titanium, and a near α Ti-5Al-2.5Sn. The size and distribution of crystallographic orientation of the α-phase, which has an HCP crystalline structure, is significant in fretting crack formation. In particular, the effect of slip displacement amplitude and number of fretting cycles on the evolution of grain morphology, grain orientation, misorientation distribution, composition, and microhardness is investigated. The fretting behavior is also related to the macroscopic monotonic and cyclic deformation response. The research goals are accomplished using state-of-the-art surface characterization tools such as orientation image microscopy (OIM) using electron backscatter diffraction (EBSD), energy dispersive X-ray analysis (EDX) and nanoindentation.

An uppermost oxidized layer is observed to form at all but the smallest slip amplitudes within 1000 cycles. An intermediate or transitional layer was observed that exhibited evidence of severe plastic deformation and layering of material. Beneath the oxidized and transitional layers, a fretting disturbed layer exists in which distinguishable grains are observed. Fretting cracks ranging in size from 10 μm to 50 μm in length are observed near the edge of contact in selected Ti-6Al-4V tests within the fretting disturbed.
layer. Crack formation is most prevalent in Ti-6Al-4V specimens when fretted at an intermediate slip displacement amplitude. Crack formation is associated with significant oxygen diffusion well into the fretting disturbed layer, as well as formation of a strong basal texture. Accumulation of cyclic plastic strain is observed in the fretting disturbed layer as indicated by cyclic hardening or softening that is consistent with macroscopic cyclic behavior.

Basal texture develops in Ti-6Al-4V and Ti-5Al-2.5Sn specimens when fretted at intermediate and higher slip amplitudes for a sufficient number of cycles. CP-Ti does not demonstrate distinct texture development with increasing slip amplitude or cycles. A strong relationship between coefficient of friction and fretting damage accumulation seems to depend on relative grain size and initial texture. Therefore, a combination of mechanisms such as plastic deformation, texture development, and oxygen diffusion observed at a critical slip amplitude play key roles in creating conditions that foster fretting crack formation.

A distinct increase in the relative frequency of low-angle misorientations for every material and test condition is observed in the fretting disturbed layer, even when more obvious visual indications of plastic deformation (i.e., cracking or flow/rotation of grain “markers”) are not observed. Therefore, the misorientation distribution is a possible quantifiable measure of fretting damage that results from an accumulation of plastic strain in this layer.

This study is the first of its kind to use OIM to characterize fretting damage and also makes contributions to the body of knowledge about deformation mechanisms in titanium alloys. The results provide a foundation for developing and validating
computational crystal plasticity models and their application to fretting and sliding contact problems. New fretting assessment measures have also been identified and have application for components that suffer from fretting wear and/or fatigue related failures.
CHAPTER I
INTRODUCTION

Components that are clamped together and are subject to a combination of fatigue loading and repeated alternating slipping over the surfaces are prone to fretting fatigue damage. Some examples include aircraft gas turbine engine components and helicopter rotor hubs, among others. Inspections of these components must be done routinely in order to avoid fretting fatigue failures, since the useful service lives are often drastically reduced compared to components subjected to plain fatigue alone. In an effort to reduce the cost of such inspections, there has been an emphasis on developing fretting fatigue prediction methods as well as damage assessment procedures that can be used to determine remaining life.

Despite the wealth of experimental test data and computational efforts that have been directed towards fretting and fretting fatigue damage prediction, the understanding of this phenomenon still remains illusive for a number of reasons. First, a global model of the crack formation process does not take into account the effects of surface conditions (roughness/asperity contact), surface modifications (shot peening, laser shock peening, burnishing, etc.), or chemical influences, other than indirectly through their influence on the friction coefficient. Hills and Nowell (1994) and Suresh (1998) have summarized a substantial portion of previous research in this area. The complexity of the surface condition effects are not reflected in existing friction models, such as Coulomb's Law,
which makes selection of an appropriate friction coefficient for analysis purposes more difficult.

The other assumptions required for a fretting and fretting fatigue damage prediction analysis, such as the material model (i.e., elastic or elastic-plastic) as well as the appropriate scale of the fatigue process volume and crack length, also have a significant impact on prediction accuracy. Recent work devoted to elastic-plastic analysis of fretting fatigue has shown some promise in illustrating the effects of cyclic plasticity and ratchetting on fretting fatigue life prediction (Ambriço and Begley, 2001) assuming non-linear kinematic hardening $J_2$ plasticity theory. However, because this model represents a homogenous material, it cannot explicitly consider microstructure.

Extremely high stress/strain gradients within a very small volume near the fretting interface occur at a depth on the order of the grain size (Swalla and Neu, 2000; Fayolle et al., 1993, Sauger et al., 2000, Goh et al., 2001). Since the stress/strain field associated with crack formation occurs at such a small scale, it is necessary to understand the role of mechanical behavior, composition, crystallographic orientation, and deformation substructure at this scale when developing computational crystal plasticity models used for predicting durability of components that suffer attachment related damage.

Titanium alloys are commonly used materials in many engineering applications in which lightweight, high strength, and corrosion resistance are of prime importance. For example, Ti-6Al-4V is used in the fan and compressor stages of gas turbines. An equiaxed α-β Ti-6Al-4V material was selected because an extensive database for this material exists for smooth specimen fatigue and fretting fatigue developed under the USAF HCF PRDA-V program and it had been used for previous fretting fatigue
experiments and computational micromechanics modeling (Wallace and Neu, 2003; Goh et al., 2001; Morrissey and McDowell, 2000)

There is arguably some consensus that fretting damage most likely results as a combination of mechanisms; those related to large plastic strains and others related to chemical interactions (i.e., diffusion). The following is a list of indicators linked to significant fretting damage and/or plastic deformation in Ti-6Al-4V (Wallace and Neu, 2003; Glaeser and Lawless, 2001; Sauger et al., 2000).

- Transgranular cracking in α grains.
- Phase change from metastable β phase to stable α phase.
- Change in grain size or shape due to large plastic strains in surface layers.
- Change in composition (i.e., diffusion of oxygen into microstructure) and transfer of material.
- Change in local mechanical properties (i.e., microhardness).

The behavior of ductile materials during sliding has received considerable attention as well (Rigney, 2000). Some of this work is relevant to the fretting problem. More work is needed to quantify complicated microstructural damage processes related to fretting crack formation such as large plastic deformation, transfer, and mechanical mixing. Recent advances in surface analysis technology have made this a realistic goal. In addition, the possibility exists to incorporate this information directly into computational models used for fretting damage prediction (e.g., Goh et al., 2001) since much of the output is computer-generated. For instance, development of instrumented indentation testing has allowed detection of changes in hardness or elastic modulus at the nanoscale and electron backscatter diffraction (EBSD) can provide a wealth of information on crystallographic orientation that was previously available only by time-consuming and painstaking diffraction measurements.
The primary focus of this research is to characterize damage at the microstructural level due to fretting by quantifying deformation processes in Ti-6Al-4V, Ti-5Al-2.5Sn, and commercially pure titanium (CP-Ti) specimens subjected to a wide range of fretting loading conditions. Displacement-controlled fretting experiments were designed to produce either partial slip or gross slip contact conditions. Using electron backscatter diffraction (EBSD), the resulting microstructure is characterized to obtain orientation maps, grain morphology, misorientation angle distribution, and texture information. Observations from EBSD experiments are coordinated with scanning electron microscopy using a high resolution field emission gun (FEG) SEM. Nanohardness of the material near the fretting contact are obtained and results are compared to the bulk material response observed in monotonic and low cycle fatigue tests.

This work is the first of its kind to use EBSD to characterize fretting damage in any material. The study of pure titanium and two titanium alloys provided a unique opportunity to more directly determine the role of the α-phase in fretting damage as well as offering more insight into HCP material behavior.

Some specific objectives of this research include:

- To use EBSD to evaluate whether a preferred crystallographic orientation, or texture, develops and also evaluate the extent of grain refinement due to fretting.
- Relate texture development with frictional behavior observed in fretting tests.
- Use EDX to determine whether oxygen diffusion plays a role in fretting crack formation.
• Determine the micromechanical behavior of the surface layers using nanoindentation and relate this to the bulk material response obtained from monotonic and low cycle fatigue (LCF) tests.

• Observe changes in misorientation distribution that may be linked to the development of a deformation substructure and relate to fretting conditions.

• Identify procedures that may be used to estimate cyclic plastic strain accumulation using the misorientation distribution.

This information is extremely valuable not only to validate computational crystal plasticity models currently being developed, but also to elucidate procedures used for damage assessment of fielded (i.e., in-service) components.
CHAPTER II
BACKGROUND

2.1 Fretting and Fretting Fatigue

2.1.1 Experimental Observations

A type of component damage known as fretting has been observed and studied for many years. Experiments were completed to study the phenomenon of fretting as early as 1927 (Waterhouse, 1972). Since then, much has been learned about the process of fretting and the mechanisms that lead to fretting induced failure. Three different terms are used to describe distinctly different fretting processes, which are fretting corrosion, fretting wear, and fretting fatigue.

Failure of components by fretting corrosion, fretting fatigue, or fretting wear display different characteristics. Waterhouse (1972) describes fretting corrosion as being “characterized by the removal of particles and subsequent formation of oxides, which are often abrasive and so increase the wear.” Fretting corrosion appears to be most damaging at lower amplitudes of slip and lower frequencies of oscillation because these conditions tend to allow more time for environmental degradation to occur on the fretting surface. Displacement amplitude is a primary factor controlling whether fretting fatigue or fretting wear dominates, as shown in Figure 2.1. Fretting wear is most often associated with higher amplitudes in the gross-slip regime, within which severe surface damage is caused by wear and is assisted by oxidation causing a loss of component fit or seizure due to
wear particle accumulation between parts. Fretting fatigue dominates when wear is no longer significant, but displacement amplitude is still present.

Failures that result from a combination of fretting and fatigue are widely believed to occur due to accelerated crack growth in the mixed stick-slip regime and are not accompanied by significant wear or oxidation. Since fatigue cracks can quickly lead to catastrophic failure, fretting fatigue is widely considered the most dangerous form of fretting damage.

Fretting fatigue is usually characterized by the presence of a small oblique crack close to the contact surface at the stick-slip boundary, which later turns direction and grows perpendicular to the applied oscillatory bulk fatigue stress. These oblique cracks have been documented to range in length from the tens of microns, which is most common, up to 3 mm (Adibazzari and Heppner, 1994; Waterhouse, 1981; Sato, et al., 1986). The formation of an oblique crack coincides with what is considered to be the nucleation phase of the fatigue life. The parameters controlling crack formation at this scale are related to the interaction of the fatigue amplitude and mean stress with factors such as normal contact pressure, tangential force, contact geometry, relative slip amplitude, and friction coefficient, which control the fretting stresses and strains. The friction coefficient is not directly controlled in fretting or fretting fatigue tests, but is influenced by microstructural features such as surface roughness, adhesion, and material texture.

Two notable programs devoted in part to understanding and attempting to predict fretting fatigue of aircraft gas turbine engines within the past few years are the U.S. Air Force High Cycle Fatigue (HCF) program and the HCF Multi-University Research
Initiative (MURI). All of the investigations in these programs studied the same pedigreed α-pol Ti-6Al-4V alloy used in the current study. A brief survey of the fretting fatigue results from these programs will be given.

Fretting fatigue has been found to significantly reduce the fatigue life of a component due to the decrease in time required to form a crack and an acceleration of the small crack growth rate compared to plain fatigue, i.e., fatigue without fretting. A comparison between smooth bar fatigue and fretting fatigue lives in Ti-6Al-4V is shown in Figure 2.2. A reduction in life by 50% or more is observed. The greatest decrease in life occurs in the high cycle fatigue (HCF) regime. In fretting fatigue tests conducted on materials such as PH13-8 Mo stainless steel, 3.5Ni-Cr-Mo-V steel and 2014A Al alloy, the fatigue strength was reduced by factors ranging between 1.5 and almost 11 times for specimens undergoing fretting fatigue compared to specimens experiencing plain fatigue alone (Lindley and Nix, 1985; Pape and Neu, 1999).

The effects of stress amplitude, relative slip amplitude and contact geometry on the pedigreed Ti-6Al-4V are shown in Figure 2.3. The stress-life relationship for fretting fatigue tends to follow a power law in the finite life regime. When gross sliding contact conditions prevail (UTRC data), the lives are greater than tests conducted when partial slip exists (Purdue and Georgia Tech data). From these tests, it appears that contact conditions in which partial slip conditions dominate throughout the cycle tend to result in lower fretting fatigue life. Also, the effect of frequency (10 Hz or 5 Hz) on fretting fatigue life appears to be minimal. However, UTRC conducted tests using a flat pad, while tests conducted at Purdue and Georgia Tech used cylindrical pads, so the influence of contact geometry cannot be ruled out. In tests conducted by Pape and Neu (1999) in
which a flat pad and a cylindrical pad were placed on either side of a fretting fatigue specimen, the primary fretting fatigue crack most often occurred first at the cylindrical pad contact site.

The influence of contact pressure on fretting fatigue life for the range of pressures considered in the HCF program were found to be relatively insignificant (Lutynski et al., 1982; Wallace and Neu, 2003). The pressure effect is significant at low contact pressures, as shown in Figure 2.4. These lower contact pressures are generally smaller than those considered in the Air Force HCF program. When the contact pressures are considerably higher, ranging within 300 to 700 MPa, the fretting fatigue life again decreases (Iyer and Mall, 1999).

SEM micrographs of subsurface damage due to fretting fatigue in pedigreed Ti-6Al-4V material are shown in Figure 2.5. Multiple cracks have formed near the trailing edge slip region of the contact. The dominant fatigue crack transitioned to a direction perpendicular to the bulk fatigue specimen loading after the crack had grown to a length of approximately 50 μm. Some cracks were arrested at grain boundaries at depths ranging from 10 to 30 μm (Wallace and Neu, 2003; Goh, et al., 2001). All of the cracks were observed to grow at angles between 40° and 60° as measured from a line perpendicular to the surface. A schematic (Figure 2.6) illustrates formation of multiple cracks along the surface of this fretting fatigue specimen.

Fretting wear, unlike fretting corrosion, is characterized by mechanical degradation of the contacting surfaces and is distinguished by the generation of significant 3rd body particles. In fretting fatigue, 3rd body particle generation is relatively small. In order to shed more light on the impact of fretting on the crack formation
process, Glaser and Lawless (2001) conducted experiments on pedigreed Ti-6Al-4V specimens that involved fretting with bulk fatigue loading (pre-fretted) and subsequently tested to failure in high cycle fatigue without fretting. The fretting slip amplitudes, $\delta_s$, considered in the test program ranged from 20 to 75 $\mu$m, which encompasses the partial slip and gross sliding regime for this material. Results showed that primary cracks ranging in length between roughly 13 $\mu$m to 90 $\mu$m were observed growing into the depth of the specimens without application of a bulk fatigue load. Interestingly, cracks were observed in every specimen tested at $\delta_s = 38 \mu$m and a normal load of 117 MPa. Cracks were observed in isolated tests conducted at other conditions, therefore, it appears that a critical threshold of slip amplitude and normal force creates a situation that favors fretting crack formation. Investigation of subsurface damage was limited to SEM micrographs of the fretting scar cross-section and energy dispersive X-ray analysis (EDX) to determine the extent of oxidation and/or diffusion at the surface. An oxide scale that appeared somewhat porous and full of microcracks was found to develop in tests with a lower contact stress of 117 MPa. Oxygen was also observed to diffuse into microcracks that had formed at the surface.

A limited number of studies that focus strictly on microstructural characterization of $\alpha+\beta$, near $\alpha$, and near $\beta$ titanium alloys subjected to fretting loads have been conducted (Fayeulle et al., 1993; Sauger et al., 2000). An oxidized layer is observed to form, and is distinguished from what is called a tribologically transformed structure (TTS) characterized using EDX and transmission electron microscopy (TEM). Microhardness measurements using a Vickers indenter indicate that hardness of the TTS layer in $\alpha+\beta$ Ti-6Al-4V and in near $\alpha$ Ti-15V-3Al-2Cr-3Sn is more than double that of
the virgin material. The TTS appears to be composed of ultra-fine, randomly oriented grains of α-Ti, regardless of the prior phase volume. Therefore, grain refinement and phase transformation from the metastable β-phase to the α-phase are considered to be two primary indicators of fretting induced microstructural changes in titanium.

The thickness of the combined oxide and TTS layer ($Z_{TOT}$) that forms due to fretting in titanium for a range of slip amplitudes was also measured (Fayeulle et al., 1993; Sauger et al., 2000), as shown in Figure 2.7. The thickness of $Z_{TOT}$ tends to saturate for a slip amplitude of 50 μm or greater. When the slip amplitude is 50 μm, for a normal force of 300N and frequency of 1 Hz, the thickness of $Z_{TOT}$ appears to stabilize between $10^3$ and $10^4$ cycles (Figure 2.8).

To summarize, it appears that the slip amplitude has a considerable effect on fretting and fretting fatigue damage. The effect of normal force has been found to have little or no effect on fretting fatigue life for the range of contact pressures most often used in gas turbine engines. The effect of frequency when tests are conducted at 10 Hz or less was also not observed to be a significant factor in fretting fatigue life. Tests in which a flat pad and cylindrical pad are fretted on the same specimen routinely show that cracks form first at the cylindrical pad site (Wallace and Neu, 2003). Therefore, the key variables are the slip amplitude and number of cycles, while the contact pressure, frequency and pad geometry are secondary.

2.1.2 Fretting Fatigue Modeling

A variety of analytical and computational methods have been used to determine the stress and strain field near a fretting contact. A thorough treatment of current analytical methods can be found in Hills and Nowell (1994), which are generally limited
to elastic behavior, and so will not be discussed here. This section is devoted to computational finite element analysis (FEA) methods used to predict fretting fatigue life because one of the primary goals of the current work is to experimentally validate the use of crystal plasticity in simulation of fretting fatigue.

Until very recently, most computational FEA of fretting damage prediction used an elastic material model. While these constitutive models are much simpler to implement and are usually more computationally efficient, this assumption overlooks the obvious evidence of significant plastic deformation that occurs at a fretting contact (Waterhouse, 2000). Clearly, for prediction of crack formation under fretting, elastic models are not physically appropriate.

Implementation of elastic-plastic material models in FEA has been used to obtain the stress/strain field near the fretting contact and input into critical plane models to estimate fretting fatigue damage (Swalla and Neu, 2001). This a useful approach for modeling fretting when the fretting damage volume is considerably larger than the critical microstructural feature controlling deformation and crack formation. As an example, the microstructure of PH13-8 Mo stainless steel is very fine, therefore, treating the material as homogeneous for modeling purposes is viable. However, materials such as Ti-6Al-4V are quite heterogeneous because the critical microstructural features, such as the grain size and phase distribution are of the same magnitude as the fretting damage volume.

The effect of material model on accumulation of plastic strain using a 2-D finite element model is shown in Figure 2.9. The crystal plasticity algorithm captures the characteristics of plastic strain accumulation observed in experiments (Goh et al., 2001; Wallace and Neu, 2003). Furthermore, a correlation between the initial crack angle
observed in Ti-6Al-4V fretting fatigue experiments (see Figure 2.5) can be observed in
the crystal plasticity model results. FEA using crystal plasticity holds the most promise
for determining the stress and strain field near the contact surface in materials like Ti-
6Al-4V that exhibit a high degree of material heterogeneity at the microstructural scale.
More in depth information concerning the derivation of the 2-D crystal plasticity
algorithm can be found in Goh (2002) and in Goh et al. (2001).

In implementing the 2-D crystal plasticity model, a number of simplifying
assumptions needed to be made, which are summarized below. A representative HCP
crystal is shown in Figure 2.10 to help illustrate these assumptions. A planar triple slip
model is used that assumes:

- Plane strain.
- Strong transverse texture.
- Primary slip mode is assumed to be solely on prismatic planes.
- Slip can occur only within X-Y plane.

Even though 2-D crystal plasticity shows promise and provides a marked contrast
to J2 models, there are clearly limitations of 2-D crystal plasticity models. 3-D crystal
plasticity for Ti alloys is currently under development partly to account for some of the
important deformation mechanisms that cannot be modeled in 2-D. Some additional
features that 3-D modeling allows are as follows:

- Microstructure with different initial texture (basal, transverse, or random) can
  be more accurately modeled.
- Development of texture due to fretting can be determined.
- Slip on other planes can be explicitly modeled as well as twinning.
• Misorientation distribution from orientation image microscopy (OIM) experiments can either be directly input into computational models, or used as verification.

2.2 Texture Analysis and Orientation Imaging Microscopy (OIM)

The vast majority of techniques for texture analysis are based on principles of diffraction of radiation by a crystal lattice (Randle and Engler, 2000). The term “texture” refers to specific preferred crystallographic orientations in a sample. In order for lattice planes to diffract radiation, it follows from Bragg’s law (Equation 2.1) that the wavelength, $\lambda$, of the radiation must be smaller than the lattice spacing, $d$, which is most often on the order of tenths of a nanometer.

$$\lambda = 2d \sin \theta$$  \hspace{1cm} (2.1)

where $\theta$ is the Bragg angle. Also, different kinds of radiation, such as neutrons, X-rays, and electrons, interact with material in different ways, which directly influences the maximum possible depth of penetration of radiation into the sample material. Each diffraction method has pros and cons and must be chosen based on the type of material and information desired. Table 2.1 compares the wavelength and penetration depths for neutron, X-ray, and electron diffraction. Light is included for comparison, though light is not diffracted by lattice planes (Randle and Engler, 2000).

Electron diffraction techniques are used with either a scanning electron microscope (SEM) or a transmission electron microscope (TEM). For X-ray diffraction, which is the most established technique for texture measurement (Randle and Engler, 2000), the texture is revealed by measuring the intensities of diffraction maxima.
Neutron diffraction is used to obtain the average macrotexture in a manner that is very similar to X-rays; however, it requires specialized facilities and so is not used as often as X-ray diffraction. When applying either X-ray or neutron diffraction, a user must sample a large continuous volume in order to obtain meaningful information because not all grains will contribute to the diffracted signal (Glavicic et al., 2003). Therefore, electron diffraction is used when microtexture information is desired.

Transmission electron microscopy (TEM) is currently the most prevalent method used to locate dislocation networks in deformed materials, which enables clear identification of deformation substructure and deformation twins. However, TEM specimen preparation and analysis is very tedious, especially if considerable spatial information is required. Therefore, TEM analysis was considered outside the scope of the current project. Also, automated methods to create orientation maps using TEM are not nearly as well developed as those for the SEM and EBSD. Because of the number of specimens and materials that needed to be analyzed, EBSD was the most appropriate tool to assess damage due to fretting. Furthermore, the relative speed in which these specimens were analyzed relative to TEM aids the development of new methods to measure deformation by allowing comparison between numerous materials. It is hoped that the current work will have relevance not only in validation of crystal plasticity models used for component design, but also to quantify damage in fielded (i.e., in-service) components that have undergone fretting or sliding. EBSD is much more amenable than TEM for use as a damage assessment tool for real-life components and structural materials.
For both SEM and TEM methods, the texture in a sample is determined by analyzing what are known as Kikuchi diffraction patterns (Kikuchi, 1928). Using TEM, Kikuchi found that electron diffraction patterns from thin films of mica formed lines whose position could be explained on geometric grounds from knowledge of Bragg diffraction from crystal planes (HKL Technology, 2001). The appearance of Kikuchi bands is created when an electron beam enters a crystalline solid, arriving at the Bragg angle $\theta_b$ at every set of lattice planes, as illustrated in Figure 2.11. These electrons then undergo elastic scattering to give a strong, reinforced beam. The locus of the diffracted radiation is the surface of a Kossel cone. Two cones of radiation result from each family of planes. When some sort of recording medium (such as a phosphor screen) is positioned to intercept the diffraction cones, a pair of Kikuchi lines appear. These lines are actually parallel conic sections, but are so straight that they appear to be lines or bands. An electron backscattered pattern (EBSP) consists of intersecting Kikuchi bands.

2.2.1 Electron Backscatter Diffraction

Electron backscatter diffraction (EBSD) using a scanning electron microscope has become the most readily used method to determine microtexture (Randle, 1992). An early report on systematic analysis of Kikuchi patterns completed by Alam et al. (1954) showed that very wide-angle patterns (up to 164\(^\circ\)) could be obtained with an apparatus consisting of an electron source, a specimen chamber, and a camera. It was found that almost no energy was lost in the scattering process, and the patterns were in good agreement with Bragg’s law.

It wasn’t until nearly 20 years later, however, that an SEM was used to provide the electron source and EBSD received its name. The patterns were obtained by tilting
the specimen and captured using a fluorescent screen hooked up to a closed circuit TV camera (Venables and Harland, 1973).

Over the years, many improvements have been made, making EBSD more accurate and convenient to use. In the late 1980's, automated EBSD pattern analysis could be completed using computer software, which made EBSD analysis a practical and efficient method to obtain microtexture information because of the rapid acquisition and indexing of Kikuchi patterns. One of the most significant hardware improvements came with the addition of a Silicon Intensified Target (SIT) low-light camera, which allowed greater sensitivity in the SEM. Imaging processing systems were subsequently introduced which allowed the live image to be frozen and a background subtraction completed in order to improve the quality of the raw EBSD pattern. In the 1990's, orientation mapping was made possible by automated stage or beam control in the SEM in conjunction with automated EBSD pattern analysis. The EBSD procedure is now capable of uninterrupted mapping of hundreds of grains in a relatively short period of time without user interaction.

A Kikuchi pattern can be quantitatively interpreted to determine the exact orientation of a single grain or even sub-grains if the grain size is much larger than the diffracting volume of the electrons producing the Kikuchi map. The diameter of the diffracting volume for EBSD measurements in most materials is on the order of 1 μm (Giavicci et al., 2003). While diffraction patterns obtained via TEM tend to be more accurate than those obtained by EBSD, the sample preparation required to do TEM work (i.e. thin foils) is considerably more difficult and time-consuming. Furthermore, only solid specimens can be prepared in this manner, which usually precludes study of
material volume around a crack, for instance. Using TEM, it can be challenging to obtain considerable spatial resolution over a comparatively large area relative to EBSD.

EBSD is used in the current research for the following reasons:

1. Microtexture information is desired.


3. Sample preparation for EBSD work is relatively easy compared to TEM.

Not only can Kikuchi patterns be used to determine orientation, as mentioned above, but qualitative evaluation of the patterns can be used to determine:

1. Grain/phase boundaries

2. Identification of certain preferred orientations, e.g. orientation of the basal plane in hexagonal close packed (HCP) structures which tends to be a preferred fracture plane in α-titanium (Wilson et al., 1997; Bache et al., 1995).

3. Lattice strain – diffuseness in the diffraction pattern is a consequence of lattice plane bending, and so can be used as a guide to determine lattice strain. Pattern quality maps have been used to determine the amount of lattice strain from pattern diffuseness (Wilkinson, 1996; Tucker et al., 2000).

In order to determine an orientation, two frames of reference (a.k.a. coordinate system) are required: one that relates to the entire specimen from which the sample is obtained and a second relating to an individual crystal. Both systems are Cartesian coordinates.

The specimen or sample coordinate system is typically chosen according to the directions associated with the forming processes experienced by the specimen, such as
rolling direction (RD), transverse direction (TD), and normal or through thickness
direction (ND). The second coordinate system is determined according to directions in
the crystal. For the case of hexagonal crystal symmetry, which is the case for HCP
material structures, the crystal directions are not orthogonal. Therefore, some orthogonal
frame needs to be associated with the crystal axes. In addition, the convention used to
describe planes and directions for the HCP structure (i.e., indices) tends to make
definition of this second coordinate system more confusing. For clarity, the method used
to determine HCP crystal indices will be discussed.

In general, a lattice plane is described according to:

$$\frac{x}{a} + \frac{y}{b} + \frac{z}{c} = 1 \quad (2.2)$$

where x, y and z are the coordinates of any point on that plane. The variables a, b, and c
are the base vectors of the unit cell, as shown in Figure 2.12. For hexagonal symmetry,
a=b=c. The reciprocal multiples of the axis intercepts, H, K, and L denote the crystal
planes. These are the Miller indices (HKL). While all crystal structures can be described
by Miller indices, a coordinate system with four axes is often used for hexagonal and
trigonal crystals leading to the description of directions and planes by a four-index
notation, known as Miller-Bravais indices (Figure 2.12). Here, three axes a1, a2, and a3
all lie at 120° to each other in the basal plane. Lattice planes are denoted by the indices
(hkil) with $h + k + l = 0$. Directions are described by the Miller-Bravais indices [uvw],
with $u + v + w = 0$. Since the third index is easily derived, it is sometimes omitted.
However, the three-index set should not be confused with the Miller indices. The
transformations linking the Miller and Miller-Bravais conventions are listed in Table 2.2
(Randle and Engler, 2000).
The crystal axes can then be made orthonormal, or in other words, normalized to be all the same length, as follows:

\[
\begin{align*}
l_{11} &= a \\
l_{12} &= b \cos \gamma \\
l_{13} &= c \cos \beta \\
l_{22} &= 0 \\
l_{23} &= b \sin \gamma \\
l_{33} &= c (1 + 2 \cos \alpha - \cos \beta \cos \gamma) / \sin \gamma \\
l_{31} &= 0 \\
l_{32} &= 0 \\
l_{33} &= c (1 + 2 \cos \alpha - \cos \beta \cos \gamma - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma) / \sin \gamma
\end{align*}
\]

(2.3)

where a, b, c are lattice parameters and \( \alpha, \beta, \) and \( \gamma \) are the interzonal angles, as shown in Figure 2.13. For hexagonal crystal symmetry, \( \alpha = \beta = 90^\circ \) and \( \gamma = 120^\circ. \) Also, \( a = b \neq c. \)

The zone axis, referenced to the crystal coordinate system, is premultiplied by a matrix \( L \) having the elements listed above. For hexagonal crystals, the transformation matrix \( L \) becomes

\[
L = \begin{bmatrix}
l_{11} & l_{12} & l_{13} \\
l_{21} & l_{22} & l_{23} \\
l_{31} & l_{32} & l_{33}
\end{bmatrix} = \begin{bmatrix}
a & -a/2 & 0 \\
0 & (a\sqrt{3})/2 & 0 \\
0 & 0 & c
\end{bmatrix}
\]

(2.4)

There are two methods that are commonly used to describe orientation. The first is by defining an orientation matrix, \( g, \) which is then referenced to the specimen coordinate system in a series of steps (shown below). The second method uses the axes or orientation of a neighboring grain (also known as misorientation) as the reference system and will be discussed shortly.

The orientation is described as the position of the crystal coordinate system \((C_a)\) relative to the specimen coordinate system \((C_s)\) as follows:
The complete matrix describing the rotation or orientation is

\[
g = \begin{bmatrix}
\cos \alpha_1 & \cos \beta_1 & \cos \gamma_1 \\
\cos \alpha_2 & \cos \beta_2 & \cos \gamma_2 \\
\cos \alpha_3 & \cos \beta_3 & \cos \gamma_3
\end{bmatrix} = \begin{bmatrix}
R_{11} & R_{12} & R_{13} \\
R_{21} & R_{22} & R_{23} \\
R_{31} & R_{32} & R_{33}
\end{bmatrix}
\]

(2.6)

The first row of the matrix are the cosines of the angles between the first crystal axis, [100], and each of the three specimen axes, X,Y,Z. The second row gives the cosine of the angles between [010] and X,Y,Z. The third row are the cosine of the angles between [001] and X,Y,Z. A more convenient and commonly used description is the Euler angles in a coordinate system known as Euler space.

The convention used here was formulated by Bunge (Bunge, 1965; 1985), illustrated in Figure 2.14. The rotations are (Randle and Engler, 2000).

1. \( \varphi_1 \) about the normal direction ND (Z-axis), transforming the transverse direction TD into TD' (Y') and the rolling direction RD (X-axis) into RD' (X').
2. \( \Phi \) about the axis RD' (rotated about X'-axis in its new orientation)
3. \( \varphi_2 \) about ND' (rotated about Z'-axis in its new orientation)

The direction cosine matrices for the three rotations are:

\[
g_{\varphi_1} = \begin{bmatrix}
\cos \varphi_1 & \sin \varphi_1 & 0 \\
-\sin \varphi_1 & \cos \varphi_1 & 0 \\
0 & 0 & 1
\end{bmatrix}, \quad g_{\Phi} = \begin{bmatrix}
1 & 0 & 0 \\
0 & \cos \Phi & \sin \Phi \\
0 & -\sin \Phi & \cos \Phi
\end{bmatrix}, \quad g_{\varphi_2} = \begin{bmatrix}
\cos \varphi_2 & \sin \varphi_2 & 0 \\
-\sin \varphi_2 & \cos \varphi_2 & 0 \\
0 & 0 & 1
\end{bmatrix}
\]

(2.7)

The rotation matrix is linked to the Euler angle matrices by the following expression when multiplied in this order:

\[
g = g_{\varphi_1} \cdot g_{\Phi} \cdot g_{\varphi_2}
\]

(2.8)
In either case, for an hexagonal crystal, the ideal orientation directions mentioned here ( [001], [010], and [001] ) are not orthogonal to the crystal unit cell.

The second choice for a reference orientation designation is to use the axes of another grain in the specimen as the reference instead of the specimen axes. This is known as the misorientation. A misorientation matrix, \( \mathbf{M} \), is calculated from the orientations of grain 1 and grain 2 (see Figure 2.15) by:

\[
\mathbf{M} = \mathbf{g}_1^{-1} \mathbf{g}_2
\]  

(2.9)

where \( \mathbf{g}_1 \) is arbitrarily chosen as the reference orientation. The angle of rotation, \( \theta \), and rotation axis, \( \mathbf{r} \), is given by:

\[
\cos \theta = (\mathbf{M}_{11} + \mathbf{M}_{22} + \mathbf{M}_{33} - 1)/2
\]

\[
\mathbf{r}_1 = \mathbf{M}_{13} - \mathbf{M}_{31}
\]

\[
\mathbf{r}_2 = \mathbf{M}_{21} - \mathbf{M}_{12}
\]

\[
\mathbf{r}_3 = \mathbf{M}_{32} - \mathbf{M}_{23}
\]

(2.10)

If \( \theta = 180^\circ \), then the rotation axis, \( \mathbf{r} \), is given by:

\[
\mathbf{r}_1 = (\mathbf{M}_{11} + 1)^{1/2}
\]

\[
\mathbf{r}_2 = (\mathbf{M}_{22} + 1)^{1/2}
\]

\[
\mathbf{r}_3 = (\mathbf{M}_{33} + 1)^{1/2}
\]

(2.11)

The angle/axis pair is a very effective way to represent the misorientation, since the description physically relates to the grain or phase boundary misorientation angle and axis.

2.2.2 Misorientation Distribution and Plastic Strain

The work of Bache et al. (1998), Wilson et al. (1997), and very recently by Salem et al. (2003) constitute a significant portion of the existing body of knowledge about
titanium gained using EBSD. Fundenberger et al. (1997) used EBSD and TEM to determine the effect of deformation on texture and drew conclusions concerning active slip systems in selected HCP materials, such as zinc, titanium, and zirconium alloys. After an extensive search of the literature, only one work was found in which the effect of fatigue loading on a near-α titanium (IMI 685 or Ti-6Al-5Zr-0.5Mo-0.25Si) was studied by observation of the misorientation distribution obtained by EBSD (Wilson et al., 1997). In the discussion that follows, the relationship between misorientation distribution and deformation behavior will be presented for FCC materials such as aluminum, copper, and nickel since the vast majority of both TEM and EBSD analyses have been conducted on these materials.

It has been routinely observed that particular substructures develop as a result of plastic deformation in FCC metals (Heilmann et al., 1983; Bay et al., 1992, Butler and McDowell, 1998; Guvenilir et al., 1998; Butler et al., 2002; Barton and Dawson, 2001; Beaudoin et al., 2000; Bhattacharya et al., 2001). These substructures constitute patterns of dislocation features (Hughes et al., 1996). These features are in the form of dislocation boundaries in medium to high stacking fault energy materials. It has been found that the average misorientation angle increases with strain and that the shape and character of the distribution of angles also evolve in strain in aluminum and nickel (Bay et al., 1992). In most of these TEM studies, there is no clear distinction about what constitutes a “grain” in terms of misorientation angles. In other words, a thorough description of dislocation boundary evolution with strain is given, and it is stated that these dislocation boundaries tend to subdivide grains. However, there is no “cutoff” in misorientation angle distinguishing a grain from a subgrain. Standard convention
dictates that a grain is defined by a misorientation of 10° to 15° (HKL Technology, Hobro, Denmark). Therefore, definitions about what does or does not constitute a grain boundary may result in some confusion when trying to relate misorientation distributions obtained from EBSD and those obtained from TEM. In order to better distinguish between the misorientation data available using either method, some terminology related to deformation substructure definition is given here. Once these substructures are better understood, assumptions concerning grain boundary definition and misorientation distribution are more easily presented.

In the work by Hughes et al. (1996) and Hansen et al. (2001), misorientations are defined as a difference in orientation between what are termed cell block boundaries and cell boundaries. Each cell block is assumed to deform by fewer active slip systems than are required to maintain compatibility, i.e. Taylor criterion, but can combine into groups that collectively fulfill the Taylor criterion. These groups of cell blocks are separated by what have been called geometrically necessary boundaries (GBBs) which include microbands (MBs) and dense dislocation walls (DDWs). Within these volumes, or groups of cell blocks, networks of loosely arranged cell boundaries called incidental dislocation boundaries (IDBs) form. A schematic illustrating the dislocation microstructure that develops due to deformation of pure aluminum cold rolled 10% is shown in Figure 2.16. It has been hypothesized that the misorientation angle distributions can be described by one scaling parameter, the average misorientation angle (Hughes et al., 1996). In this study, the maximum misorientation angle used in the analysis was restricted to a maximum of 62.8° due to cubic symmetry. It may be assumed, therefore, that nearly all misorientations within a particular area chosen for
scanning using TEM were considered in the analysis—not just low-angle misorientations (i.e. less than 10°). Using this method, the average values represented by the misorientation distribution scale with strain for a particular type of cell structure (Figure 2.17). For GN Bs, the average angle varies roughly as a 2/3 power with strain, and as the square root of strain for DBs.

In the study described above, and in most work of this nature, TEM is used to explicitly determine the type of cell boundary that forms due to deformation. EBSD is not capable of determining dislocation networks. However, the existence of subgrains can be indirectly observed via the distribution of misorientation angles. Gorynin et al. (1988) concluded that the substructure morphology of polycrystalline pure α-phase titanium subjected to uniaxial tension is similar to FCC and BCC metals once a critical strain threshold of about 0.4 is reached.

Based on data obtained from EBSD scans on OFHC copper, Barton and Dawson (2001) came to the conclusion that “the misorientation angle is the natural scalar measure of the distance between orientations” and subsequently defined an average orientation based on minimizing the weighted sum of the squares of the misorientation angles within a grain. Finite-element simulations of a two-phase titanium alloy were completed by Barton and Dawson (2001), but no attempt was made to verify their numerical formulation with actual EBSD scans of titanium. The cell type is not distinguished in this case, however, they also define a grain boundary as a misorientation of 10° between nearest neighbors. The Barton and Dawson (2001) method does not rely on explicit identification of cell type. Therefore, their method appears to be more amenable to incorporation into FEM than the model proposed by Hughes et al. (1996). However, the
simple relationship between misorientation angle and strain developed by Hughes et al. (1996) appears to be more useful when directly comparing experimental tests and computational crystal plasticity analyses. It is also very useful for rationalizing the increase in relative frequency of low angle misorientations (<10°) routinely observed in deformed metals.

In Figure 2.18 (a) and (b), the relative frequency of misorientation angles distinguishing GNBs and IDBs observed using TEM in pure nickel cold rolled to a reduction of 98% are plotted, respectively. The misorientation angle of IDBs is consistently less than 10°, while GNBs exhibit a relatively high frequency of misorientation angles across a much wider spectrum, including angles greater than those traditionally used to define grain boundaries. In other words, there is some confusion whether their classification of a GNB is a subgrain or what could be considered a grain, if one chooses to use a threshold value of misorientation angle (i.e. 10°) as the definition of a grain. In the EBSD analyses that follow, a grain boundary will always be defined as having misorientations greater than 10°, so any misorientations less than 10° are assumed to define intergranular misorientations. This chosen angle is somewhat subjective, as some use an angle as high as 15° to define a grain. Although EBSD cannot be used to obtain explicit knowledge of the exact type of cell or subgrain structure, a wealth of information can be derived from plotting the misorientation angle distribution for a given sample.

Channel 5 software (HKL Technology) provides a method by which the misorientation data can be plotted as a histogram of relative frequency versus misorientation angle, an example of which is shown in Figure 2.19. If the data has been
plotted correctly, the sum of the relative frequencies of all angles, divided by the number of bins, then multiplied by 100% should equal 1. The dark line on the plot represents the theoretical random distribution for an HCP crystal. In this case, all of the misorientation angles are given, not just those that may be intra-granular in nature. When the overall distribution is compared to the theoretical random distribution, some measure of texture can be observed. Observation of misorientation angles above the angle used to define a typical grain (typically 10° or 15°) also provides clues to possible constraints between grains that may affect the deformation behavior leading to crack formation.

The misorientation distribution obtained by using EBSD has been used to determine the amount of strain in 304L and 316L low carbon stainless steel (Angeliu et al., 2000). A threshold value of 10° was used to define a grain, after which all misorientations within a particular grain were then averaged. Using this procedure, the average intergranular (a.k.a. intra-grain) misorientation scaled linearly with percent tensile strain, as shown in Figure 2.20. The 300 series of stainless steels are considered to be low stacking fault materials (Rainforth et al., 1992). Therefore, it appears that the average intra-grain misorientation angle may be used to correlate with strain, regardless of the material SFE, if the strain is not too large, say, less than 20 or 30%.

The procedure used by Angeliu et al. (2000) may be used to evaluate the average intra-grain misorientation in the titanium alloys studied in the current work, especially considering the fact that they also used Channel 5 software (HKL Technology) to conduct EBSD analysis. This effort was considered to be outside the scope of the current work, but will be considered for future research. The goal in the current work was not to explicitly define a method to predict strain, but to compare and contrast deformation
mechanisms that occur in Ti-6Al-4V, Ti-5Al-2.5Sn and CP-Ti in order to make more informed judgments concerning crystal plasticity model assumptions and damage assessment methods.

2.2.3 Microtexture Evolution

Of particular interest in fretting research are studies relating the texture of materials subjected to sliding with the evolution of friction coefficient, as well as the possible development of texture that may lead to reorientation of grains into a configuration more favorable for crack formation.

Initial texture in titanium has been shown to have an effect on the mechanical properties (Bache and Evans, 2001; Lütjering, 1998; Peters et al., 1984). Pole figures illustrating typical texture in titanium are shown in Figure 2.21. The convention for pole figures of HCP crystals have the [0001] direction, which is the normal to the basal plane, oriented parallel to the Z(ND) direction in a specimen. Therefore, a pure basal texture is one in which the angle between the basal plane normal and the Z(ND) direction is 0°. However, specimens with poles oriented away from 0° are often referred to as having a basal texture if the angle is within roughly 45° and centered at Z(ND), similar to Figure 2.21 (a). A schematic showing the orientation of the HCP unit cell with respect to the plate is shown just to the right of the pole figure.

A pure transverse texture is one in which the HCP basal plane normal is oriented towards either the X(RD) or Y(TD) direction. In other words, the basal plane normal is oriented at exactly 90° from the Z(ND) direction. For practical purposes, the poles can be oriented within about 65° from the X(RD) or Y(TD) direction and still be considered a
transverse texture (Figure 2.21 (b)). A combined basal/transverse texture has poles oriented in both directions described above. An example is provided in Figure 2.21 (c).

Of particular interest in the current study is the effect of texture on friction coefficient. In computational finite element analysis used for fretting, the friction coefficient is the one variable often used to account for a range of surface conditions. Therefore, changes in friction coefficient greatly impact deformation response and life prediction.

The contribution of crystallographic texturing on the sliding friction behavior of an α-titanium has been studied by Farhat (2001). In this study, two groups of HCP titanium samples were prepared and the effect of sliding on friction coefficient and grain reorientation were measured for each. One set of samples had an initial basal texture in which the poles were oriented normal to the sliding surface with planes oriented parallel to the surface. The second group of samples had an initial transverse texture in which the basal planes were oriented perpendicular to the sliding surface.

Results of tests on the samples with initial basal texture indicated that the friction coefficient sharply increased to a value of about 0.69 within a sliding distance of 10 m, then remained relatively constant afterwards, as shown in Figure 2.22 (a). In these samples, it was shown that the initial basal texture was not significantly altered by sliding.

The effect of sliding on the friction coefficient for the samples with initial transverse texture (Figure 2.22 (b)) indicates markedly different behavior than the samples with initial basal texture. In this case, the friction coefficient increased to a peak of 1.04 followed by a transition to a steady state value of 0.59. A basal texture developed
with increasing sliding distance in the samples with initial transverse texture. The drop in friction coefficient with sliding in the transverse samples is attributed to reorientation of the basal planes in a direction parallel to the sliding surface. Others have also reported development of basal texture with sliding in HCP titanium (Scott and Wilman, 1958). It is believed that crystallographic reorientation can produce a reduction in the effective shear-stress in near surface regions and a subsequent reduction in the friction force required to maintain relative motion (Kuhlmann-Wilsdorf, 1980). In summary, plasticity of the near surface layers plays a key role in determining the friction coefficient.

2.3 Deformation at Contacts

The body of knowledge concerning wear of materials is extensive. While it is beyond the scope of the current research to offer a thorough review of wear processes due to sliding, selected examples are presented here in order to provide insight into microstructural changes that occur due to fretting, especially in the fretting wear regime. Also covered are models developed to rationalize changes in mechanical properties, composition, and grain morphology that occur near the surface due to sliding.

Many changes have been observed to occur in sliding contact conditions in which there is an absence of effective lubrication. Rigney (2000) categorizes microstructural changes due to plastic deformation and those due to other factors in two broad classifications. The first classification is one in which the changes are considered to be independent of the choice of counterface material where plastic deformation, shear instabilities, deformation substructure, crystallographic texture, phase transformations and fracture may all be relevant. The second classification involves changes where the
environment is important, i.e. those factors related to adhesion and chemical reactivity (e.g., oxidation). Mechanical alloying, ball milling, and nanocrystalline materials are considered to be a part of the second group (Rigney, 2000).

Specimens that have been subjected to sliding loads often exhibit evidence of extreme plastic flow in the surface layers in the form of flow patterns and rotation of "marks" in the near surface microstructure. Suitably oriented grain boundaries, twins, or lamellae can function as a marker, and can be used to estimate strain and strain rates (Rigney, 2000; Rainforth, 2001). Assuming the shear deformation is parallel to the surface and neglecting contributions from compression and rotation of the surface layers, the following equation has been used to estimate strain \( \varepsilon \) due to sliding:

\[
\varepsilon = \frac{\sqrt{3}}{3} \tan \theta
\]

(2.12)

where \( \theta \) is the shear angle between grain boundaries and the normal to the worn surface. (Dautzenberg and Zaat, 1973; Moore et al., 1976; Perrin and Rainforth, 1997; Hughes et al., 1995).

The evolution of deformation substructures that result from sliding is almost exclusively focused on FCC materials such as copper, aluminum, and nickel. These materials tend to follow a well-defined sequence of microstructural changes as the strain is increased. For example, in TEM images of an OFHC copper block that has been slid against a 440C steel ring, one can detect a gradual change from loosely organized groups of dislocations, to cell, and then to subgrains as the surface is approached (Heilmann et al., 1982). This test was conducted in an argon environment with 20% relative humidity. A schematic representing patterns of cells that form due to sliding is shown in Figure 2.23. The misorientation angle increases as the surface is approached. Cells or subgrains
are separated by low-angle boundaries far away from the surface. Closer to the surface, misorientations as high as 20° are observed. At the very top layer, a nanocrystalline layer that was influenced by the counterface material and the environment is observed (Ganapathi et al., 1990).

Of some interest for fretting applications are studies showing a dependence of surface hardening or softening on the stacking fault energy SFE (Hirth and Rigney, 1976). The delamination theory of wear rationalizes that a soft surface layer on the scale of about 1-3 grains or about 10 μm results from dislocation removal from the very near surface region under the action of image forces (Suh, 1980). However, it was proposed by Hirth and Rigney (1976) that compatibility constraints are more likely the cause of a soft surface layer at the 10 μm scale. The cells in high SFE materials tend to be strong barriers to dislocation mobility and resemble grains, so the scale at which image forces may play a role is expected to be much smaller (i.e. between 0.1 and 1 μm) in high SFE materials than it is in low SFE materials. Interestingly, the materials with high SFE were predicted to always harden at a scale within 10 μm from the worn surface, with image force effects playing a role at the scale of 0.1 to 1 μm.

If compatibility effects dominate near surface deformation behavior, then a strong case can be made for using a crystal plasticity algorithm because this type of formulation is explicitly designed to take compatibility effects into account. Homogeneous material models, by definition, cannot consider compatibility between individual grains and constraints due to orientation of slip systems.

The number of available slip systems in a material is another factor influencing compatibility effects. In cubic materials, there is more tendency for a transition to
multiple slip than with hexagonal metals, which have a limited number of available slip systems. The slip system most active in HCP materials is dependent upon the c/a ratio. Most HCP metals with c/a ratios less than ideal, such as titanium, slip primarily occurs on prismatic planes. Those with nearly ideal or higher than ideal c/a ratios tend to slip primarily on the basal plane. More detail concerning deformation mechanisms in HCP titanium can be found in Chapter 3. It should be noted that HCP metals with less than ideal c/a ratios, such as titanium, are known to have serious seizing or adhesion problems except when coatings are present (Hirth and Rigney, 1976).

Another factor tied to hardness or softness at surfaces subjected to sliding is the size of the grains or cells. The Hall-Petch relation of the form \( \sigma_f \propto d^{-1/2} \) implies that the flow stress, \( \sigma_f \), increases with decreasing cell or grain size, \( d \). In turn, the depth of the surface hardening or softening region (previously estimated to be one to three grain or cell diameters) is expected to scale directly with the cell or grain size (Hirth and Rigney, 1976).

A model proposed by Hughes et al. (1995) is also of a form similar to the well-known Hall-Petch equation:

\[
\sigma_f = \sigma_s + \frac{KGb}{D^2}
\]  

(2.13)

where \( \sigma_f \) is the flow stress, \( \sigma_s \) is the frictional stress, \( K \) is a non-dimensional material constant which typically has the value of 20 for metals and alloys (Rainforth, 2000), \( G \) is the shear modulus, \( b \) the burgers vector, \( D \) the subgrain size, and the exponent \( n \), varies empirically from 0.5 to unity. In the work by Hughes et al. (1995), the value of the exponent \( n \) between 0.5 and unity was used as bounding values in order to make flow stress estimates. This model was preferred over those that rely on explicit dislocation
density, especially in materials such as copper that tend to exhibit inhomogeneous dislocation arrangements in cell and subgrain boundaries. This argument may be a convenient way to avoid having to consider the effect of SFE on dislocation ordering.

A value of unity is considered typical for dislocation cells and a value of 0.5 is used for subgrains. The familiar Hall-Petch relationship (n = 0.5) is more appropriate if one assumes that subgrain boundaries are assumed to act as dislocation barriers (Rainforth, 2000). Whether the grain or subgrain diameter is inserted into equations of this form seem to be a matter of individual interpretation and solely dependent upon the scale of interest. There is some scale at which it is anticipated that the Hall-Petch relation breaks down. In essence, the Hall-Petch relation predicts a bow stress that tends to infinity as the grain size goes to zero. For some metals with grain sizes on the order of a few tens of nanometers, a new regime appears, whose quantitative description is still a matter of research (Van Swygenhoven et al., 1999; El-Sherik et al., 1992).
### Table 2.1: Comparison of diffraction sources.

<table>
<thead>
<tr>
<th></th>
<th>Light</th>
<th>Neutron</th>
<th>X-rays</th>
<th>Electrons</th>
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</thead>
<tbody>
<tr>
<td>Wavelength (nm)</td>
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<td>0.05-0.3</td>
<td>0.05-0.3</td>
<td>0.001-0.01</td>
</tr>
<tr>
<td>Penetration depth, absorption length (mm)</td>
<td>---</td>
<td>10-100</td>
<td>0.01-0.1</td>
<td>10⁻³</td>
</tr>
</tbody>
</table>

### Table 2.2: Transformation between Miller (HKL) and Miller-Bravais (hklf) conventions.

<table>
<thead>
<tr>
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<th>(HKL) ⇒ (hklf)</th>
<th>(hklf) ⇒ (HKL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Planes</td>
<td>H=h; K=k; L=l</td>
<td>h=H; k=K; l=-H-K; l=L</td>
</tr>
<tr>
<td>Directions</td>
<td>U=u; V=ν; W=w</td>
<td>u=2U-V; v=2V-U; t=-U-V; w=3W</td>
</tr>
</tbody>
</table>
Figure 2.1: Illustration of variation in fretting fatigue life and fretting wear rate with displacement amplitude (Vingsbo and Söderberg, 1988).

Figure 2.2: Comparison of fretting fatigue lives versus smooth non-fretting fatigue lives for Ti-6Al-4V (f=20 Hz, flat contact with pressure = 200 MPa) (Anton, 1999).
Figure 2.3: Comparison of Ti-6Al-4V fretting fatigue test results from UTRC, Purdue, and Georgia Tech (Wallace, 2002).

Figure 2.4: Effect of contact pressure on the fretting fatigue life of Ti-6Al-4V (f=20 Hz, $\sigma_a = 150$ MPa, R=0.1, with flat contact geometry) (Nakazawa et al., 1992).
Figure 2.5: SEM micrograph showing subsurface region of the lower cylindrical contact scar of specimen 113-S323 (170,000 cycles at $\sigma_{\text{max}}=150$ MPa, $R=0.1$, $p_{\text{ac}}=70$ MPa, $p_{\text{eff}}=348$ MPa) showing (a) unetched cross-section with dominant fatigue crack and multiple smaller surface cracks, (b) three nonpropagating cracks one of which has stopped at an alpha grain boundary, and (c) an etched view of the entire contact region showing the distribution of alpha and beta phase in the fretting affected region (Wallace and Neu, 2003).
Figure 2.6: Schematic of the subsurface contact and cracking behavior at the lower cylindrical contact scar in specimen 113-S323 (170,000 cycles at $\sigma_a=150$ MPa, $R=0.1$, $p_{min}=76$ MPa, $p_{avg}=348$ MPa) showing (a) the stick, slip, leading edge, and trailing edge regions and (b) crack initiation sites and growth directions within the trailing edge region (Wallace, 2000).
Figure 2.7: Variation in combined oxide and TTS layer ($Z_{TOT}$) versus displacement for $\alpha + \beta$ phase titanium (TA6V), $\alpha$-phase titanium (TV15CA) and $\beta$-phase titanium (TV15CA) (Sauger et al., 2000).

Figure 2.8: Variation in combined oxide and TTS layer ($Z_{TOT}$) thickness versus number of cycles for an $\alpha + \beta$ phase titanium (TA6V), $\alpha$-phase titanium (TV15CA) and $\beta$-phase titanium (TV15CA) tested at $\delta_s = 50 \mu m$, 300N normal force, and $f=1$ Hz (Sauger et al., 2000).
Figure 2.9: Comparison of accumulation of plastic strain between 2-D finite element analysis of fretting fatigue using (a) J2 nonlinear kinematic hardening plasticity model and (b) crystal plasticity model after three cycles (Goh et al., 2001).

Figure 2.10: Two dimensional representation of prismatic slip in HCP structure (Goh et al., 2001).
Figure 2.11: Origin of Kikuchi lines from the EBSD perspective (Randle and Engler, 2000).

Figure 2.12: Planes and directions in the (0001) basal plane of a hexagonal crystal. Both Miller and Miller-Bravais indices are shown (Randle and Engler, 2000).
Figure 2.13: Orthonormalized crystal coordinate system for a) hexagonal and b) general symmetries.
Figure 2.14: Schematics illustrating Bunge convention for Euler angle rotation (a) relative to crystal orientation and specimen directions (b) representative specimen (Randle and Engler, 2000).
Figure 2.15: Diagram showing the angle/axis rotation between two hexagonal grains (adapted from Vicens et al., 1994).

Figure 2.16: Schematic illustrating microstructure found using TEM for pure aluminum cold-rolled 10%. Rolling direction is marked by RD. The GNBs are marked A, B, C, ...; the IDBs are marked a, b, c, ...; cell blocks are marked CB1, CB2, CB3, CB4, CB5, CB6 (Hughes et al., 1996).
Figure 2.17: Power law relationships between the average misorientation angles and the applied strain (Hughes et al., 1996).
Figure 2.18: Histogram showing the distribution of misorientation angles across lamellar boundaries in pure nickel (99.99%) cold rolled to a reduction of 98% (b) similar to (a) but showing angles across cell boundaries lying in the volume between the lamellar boundaries (Hansen et al., 2001).
Figure 2.19: Example of relative frequency versus misorientation angle distribution histogram plot provided by Channel 5 software. Theoretical random distribution for an HCP material is plotted as a dark line for comparison.

Figure 2.20: The average intra-grain misorientation for 304L and 316L stainless steel tensile specimens strained 1,2,5,10,15 and 20% in tension at room temperature (Angeliu et al., 2000).

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Figure 2.21: Texture in Ti-6Al-4V produced by differences in temperature and rolling direction during manufacturing (a) basal produced by cross rolling at 800°C (b) transverse produced by unidirectional rolling at 960°C and (c) basal/transverse produced by unidirectional rolling at 800°C (Peters et al., 1984).
Figure 2.22: Coefficient of friction versus sliding distance curves obtained using a normal load of 1.0 N and a sliding speed of \(1.3 \times 10^{-1} \text{m/s}\) under unlubricated sliding condition for (a) HCP titanium with initial basal or "normal" texture and (b) HCP titanium with initial transverse texture (Farhat, 2001).
Figure 2.23: Schematic showing pattern of cells observed using TEM of OFHC copper slid against 440C steel ring. The numbers illustrate misorientation in degrees across cell boundaries. Areas with similar shading are associated with same parent grain (Heilmann et al., 1983).
CHAPTER III
MATERIAL BEHAVIOR

3.1 Materials and Microstructure

Fretting tests were conducted on commercially pure (CP) titanium and two titanium alloys, Ti-6Al-4V and Ti-5Al-2.5Sn. The composition of each material is listed in Table 3.1. CP-Ti Grade 1 was obtained as a 0.75" x 96" x 253.75" rolled plate from Titanium Industries, Parsippany, NJ. The Ti-5Al-2.5Sn was obtained in a 2 ¼" diameter x 9 ¾" long rolled bar from President Titanium, Boston, MA. The Ti-6Al-4V was obtained as a forged plate, labeled #113 from the Air Force HCF program, with dimensions approximately equal to 16" x 6" x 0.80".

Both the CP-Ti and Ti-5Al-2.5Sn have equiaxed microstructures with a mean grain diameter approximately equal to 55 μm and 25 μm, respectively. The mean grain diameter was determined with image recognition software using a line intercept method per ASTM E112-96. An optical image of CP-Ti after etching with 5% hydrofluoric acid (HF) was obtained with an Olympus BX40 microscope with cross-polarized light filter and is shown in Figure 3.1. The microstructure is pure α-phase.

An optical image of Ti-5Al-2.5Sn after etching with 0.5% HF is shown in Figure 3.2. Ti-5Al-2.5Sn is considered to be a near α-phase titanium alloy due to the presence of a small amount of globular β phase particles interspersed within the α phase grains.
The Ti-6Al-4V is a bimodal, duplex microstructure consisting of 60% α and 40% β phase, as shown in Figure 3.3. Characteristic of a bimodal microstructure is the large, nearly equiaxed α phase grains randomly dispersed with grains consisting of lamellar α + β phases. The term “duplex” refers to the duplex annealing heat treatment used to obtain this microstructure (Donachie, 2000).

Mechanical property information for Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti can be found in Chapter 5.

3.2 Deformation Mechanisms in Titanium

This section is devoted to background information concerning the most commonly observed deformation mechanisms and factors that control both macroscopic and microscopic mechanical response in single crystal and polycrystalline CP-Ti, Ti-5Al-2.5Sn, and Ti-6Al-4V. The materials are presented in order of complexity and amount of alloying.

3.2.1 CP-Ti

CP-Ti is commonly available in four grades (CP1-CP4). Each grade is primarily distinguished by the amount of iron and oxygen impurities. CP Grade 1, which is the grade obtained for this study, is considered the purest. In pure titanium, the α-phase is characterized by a hexagonal close-packed (HCP) crystalline structure and is stable from room temperature to approximately 882°C (1620°F). Two trace alloying elements, oxygen and iron, most directly affect the CP-Ti material properties and microstructure. Grades of higher purity are lower in strength and hardness than those with a higher interstitial content. Oxygen tends to stabilize the α-phase, i.e. raise the temperature at
which the alloy will be transformed completely to the $\beta$-phase. This temperature is known as the beta-transus temperature. The beta-transus is critical in deformation processing and in heat treatment. Iron stabilizes the $\beta$-phase by lowering the temperature of transformation (Donachie, 2000). However, there are only trace amounts of iron in CP-Ti, so the material is considered to be pure $\alpha$-phase. The main effect of iron in CP-Ti is limiting grain growth in subsequent heat treatments.

The $\beta$-phase in titanium is a body centered cubic (BCC) structure. Other alloying elements, such as vanadium, molybdenum, chromium, and copper are also $\beta$-phase stabilizers, the effects of which will be discussed in the section focused on Ti-6Al-4V (Section 3.2.3).

In HCP materials, crystal lattice reorientation due to deformation is primarily caused by mechanisms such as dislocation glide along slip planes and by deformation twinning. Deformation twinning and slip are competing mechanisms in HCP materials. The most common slip planes and slip plane directions in HCP titanium are illustrated in Figure 3.4. Slip in the HCP $\alpha$ phase occurs on $\{10\overline{1}0\}$ prismatic planes, $\{10\overline{1}1\}$ first order pyramidal planes, and the (0001) basal plane. The differences associated with slip deformation and deformation twinning are illustrated in Figure 3.5. The twinning process (Figure 3.5(c)) results in a rotation of the lattice such that the atom positions in the twin represent a mirror image of those in the untwinned material. Alternatively, slip occurs by translations along widely spaced planes such that the relative orientation of different regions in the slipped cube remains unchanged, as shown in Figure 3.5 (b). While the maximum potential elongation of HCP metals due to twinning is small compared to slip, the rotation of the crystal within the twin serves to reorient the slip planes so that they

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experience a higher resolved shear stress, which contributes to more deformation by slip processes. HCP crystals deform by \( \{1124\}, \{1122\}, \) and \( \{10\overline{1}1\} \) twins during compression along the c-axis, and by \( \{10\overline{1}2\}, \{1123\}, \) and \( \{121\} \) twins during tensile loading along the c-axis (Suresh, 1998). The most common twinning planes are shown in Figure 3.6.

In high-purity titanium, \( \{10\overline{1}0\} \) slip predominates under both monotonic and cyclic loading at room temperature (Nemat-Nasser et al., 1999; Tan et al., 1998; Fundenburger et al., 1997). The effect of temperature and strain rate on twin number density in CP-Ti is shown in Figure 3.7 (Nemat-Nasser et al., 1999). Twinning in titanium is favored over slip deformation only at low temperatures, high strain rates, and in materials with high-purity and relatively coarse grain size (Meyers, 1999; Russo and Seagle, 1994). Twinning is not favored in Ti-6Al-4V or in Ti-5Al-2.5Sn at the strain rate and temperatures in which the fretting fatigue experiments are conducted, which is attributed to the addition of aluminum. However, it is likely to be a significant factor in the deformation of CP-Ti due to its purity as well as the grain size of the sample used in the fretting tests.

CP-Ti strain hardens slightly under cyclic loading, and exhibits a small amount of strain hardening in monotonic tests. Single crystal and polycrystalline material response is believed to depend on the extent of twinning. The cyclic stress-strain behavior of randomly oriented single crystals of high-purity titanium subjected to multiple-step fatigue testing using a method developed by Landgraf et al., (1969) were found to fall into three groups depending on orientation and the extent of slip and twinning (Gu et al., 1994). These experiments, as well as others conducted on \( \alpha \)-Ti single crystals indicate
that the cyclic deformation is strongly influenced by the propensity for twin formation, which is illustrated in Figure 3.8 (a). Here, a marked increase in the cyclic hardening rate coincides with an increase in the occurrence of cyclic twins. The unit triangle in Figure 3.8 (b) shows the orientation of the specimens A, B, and C. Specimen A exhibited the most deformation due to twinning, while specimen B exhibited the least amount of twinning. A thorough review of mechanical twinning theory is provided by Reed-Hill (1964), Hertzberg (1983), and Meyers (1999).

At low tensile strain (i.e. \( \varepsilon = 0.1 \)), the dislocation distribution of polycrystalline CP-Ti reveals a considerable spatial non-uniformity which results in low-angle boundaries (Gorynin et al., 1988). Within increasing strain, the crystals tend to “fragment”. Fragmentation intensifies with deformation, with dislocations becoming more localized in bands separating equidistant parallel volumes. Misorientations between these fragments grow with increasing strain. As the misorientation angles increase, the morphology of the fragmented structure of \( \alpha \)-Ti is similar to that observed for FCC and BCC metals. Specifically, “transverse boundaries which divide the initial band structure into equiaxial fine microregions appear inside elongated bands” (Gorynin et al., 1988). The deformation substructure of cold-rolled FCC aluminum is similarly described by Hughes et al. (1996) and Hansen et al. (2001). Gu et al. (1994) also conclude that fatigued \( \alpha \)-Ti single crystals exhibit substructure evolution similar to fatigued FCC crystals.

Identification of twins in compression tested CP-Ti specimens has been accomplished using EBSD analysis (Salem et al., 2003). The misorientation angle plot and orientation map is shown in Figure 3.9 (a) and (b) respectively. In this study, a
misorientation angle of 63°, with a rotation axis corresponding to the \( \{11\bar{2}2\} \) family of compressive twins confirmed visual observations of twins found using SEM.

The thermal conductivity of titanium at room temperature is low relative to many other metallic elements, so the frictional heat generated by fretting does not dissipate as readily in titanium as it would in other metals. The addition of alloying elements further reduces thermal conductivity. For example, the thermal conductivity of most titanium alloys is half that of stainless steels and one tenth that of aluminum alloys (Seagle and Russo, 1988). Another factor to consider is the fact that titanium is known to readily absorb embrittling interstitial elements such as oxygen, nitrogen, and hydrogen. However, the temperature at which titanium becomes chemically active is about 650°C (1200°F), so this is mainly a concern during heat treatment. The build up of an oxide layer within a small volume near the fretting interface may result due to heat generation and mechanical mixing from large plastic deformation.

3.2.2 Ti-5Al-2.5Sn

Ti-5Al-2.5Sn is a near-\( \alpha \) titanium and is one of the first titanium alloys to be developed commercially. There are very low concentrations of \( \beta \) stabilizers (i.e. iron) in Ti-5Al-2.5Sn. Both of the alloying elements (aluminum and tin) are \( \alpha \) stabilizers. Tin has extensive solid solubility in both the \( \alpha \) and \( \beta \) phases, so it acts as a strengthening agent.

Ti-5Al-2.5Sn is most often used in cryogenic applications due to its ability to retain ductility and toughness at very low temperatures. In particular, Ti-5Al-2.5Sn has superior fracture toughness at both room temperature and elevated temperature in comparison with Ti-6Al-4V (Fifthal et al., 1978). However, it has lower stress-corrosion

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resistance than other commonly used titanium alloys. The monotonic and cyclic stress-strain behavior is similar to that of Ti-6Al-4V.

TEM studies of Ti-5Al-2.5Sn specimens subjected to tensile loading and fatigue loading have recently been conducted (Leguey et al., 2002). Under tensile loading, a cellular dislocation structure was observed near the grain boundaries, and a high density of long parallel dislocations were also observed. The main gliding planes were the prismatic and pyramidal planes. Grains with different orientations tended to activate preferential slip on basal planes. The three slip modes identified are \{\{0\overline{1}0\}\} <1\overline{2}\overline{1}0>, \{0001\} <1\overline{2}\overline{1}0>, and \{1\overline{0}\overline{1}1\} <1\overline{2}\overline{1}0>. No twins were detected, and the majority of the dislocations were nearly pure screw character. In particular, Ti-5Al-2.5Sn was chosen as a study material because the dominant deformation mechanisms observed in the \(\alpha\) grains are similar to the \(\alpha\)-phase in the more complex bimodal Ti-6Al-4V. Also, similar to Ti-6Al-4V, the Ti-5Al-2.5Sn it does not tend to twin.

In fatigued specimens, the \(\alpha\) grains appeared to be divided by subgrain boundaries with a high density of entangled dislocations and small defects in the subgrains. Tilting experiments showed that most of these defects were a-type. Basal slip bands were observed in a single grain, while bands of common prismatic planes were observed in contiguous grains. In fact, simultaneous activation of both prismatic and basal systems were observed in the same grain. The small defects appeared to be grouped in a layered structure that follows prismatic slip planes. These layers were separated by nearly defect-free regions, leading to the conclusion that strain is strongly localized during the fatigue process (Leguey et al., 2002).
Microstructure has been shown to influence the crack path of fatigued near-α titanium (Ti-6Al-5Zr-0.5Mo-0.25Si) (Wilson et al., 1997; Bache et al., 1998). In this case, the size and crystallographic orientation of α-colonies is the controlling element. Colonies are regions within prior β-phase grains (PBG) that have α-platelets with nearly identical orientations. The orientation relationship between a colony and a PBG is controlled by the Burgers relation.

\[
[\{110\}_b // (0001)_a, \langle 111 \rangle_b \parallel \langle 11\overline{2}0 \rangle_a]
\]

(3.1)

If colonies obey the Burgers relation, then PBG sharing colonies will have basal planes at angles of 0°, 60°, and 90° in the ratio of 1:4:1 (Wilson et al., 1997). EBSD measurement of misorientations between PBG sharing colonies located near a fatigue fractured surface indicated an increase in the proportion of misorientations between 57.5° and 62.5°. The proportion in the overall distribution of angles changed to 5:8:3, which they suggest is a representation of an increase in common basal planes for fractured specimens.

3.2.3 Ti-6Al-4V

Ti-6Al-4V is the most widely used Ti alloy, accounting for 50% of all the titanium used in the world. More than 80% of Ti-6Al-4V usage is by the aerospace industry. The next biggest user is the medical prosthesis industry (Donachie, 2000).

As mentioned above, aluminum is an α-phase stabilizer while vanadium is a β-phase stabilizer. Vanadium falls within the β isomorphous group of β stabilizers, meaning that it is miscible in the β phase.

The duplex heat treated Ti-6Al-4V studied here tends to cyclically soften and exhibits nearly elastic-perfectly plastic material response. TEM studies of Ti-6Al-4V
specimens subjected to tensile loading and fatigue loading have been conducted (Leguey et al., 2000). The microstructure of the tensile tested Ti-6Al-4V specimens were similar to the tensile test Ti-5Al-2.5Sn specimens discussed in the previous section. A high density of long parallel \( <a > \) type dislocations are observed in regions where deformation induced subgrains can be seen. Electron diffraction verified that a martensitic transformation from the metastable \( \beta \) phase to the \( \alpha' \) (martensite) phase occurred.

In fatigue tests, the same behavior was observed in the \( \alpha \)-phase for both the Ti-6Al-4V and the Ti-5Al-2.5Sn (see Section 3.2.2). The \( \alpha \) grains are subdivided by subgrain boundaries with a high density of entangled dislocations and small defects in the subgrains (Leguey et al., 2002). The misorientation angles between subgrains were not documented.

A considerable amount of research has been devoted to determining which slip planes are most active in Ti-6Al-4V. The general consensus is that the most favored slip systems, in order of activation, occur on prismatic, basal, and then pyramidal planes (Feagus et al., 1997a; Zaeflerer, 2003; Yoo, 1981; Fundenburger et al., 1997). Twinning is not observed in Ti-6Al-4V.
Table 3.1: Composition of materials used in fretting tests.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Heat Treat</th>
<th>N</th>
<th>Al</th>
<th>V</th>
<th>Sn</th>
<th>C</th>
<th>H</th>
<th>Fe</th>
<th>O</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP 1</td>
<td>Ti-BN44 23C</td>
<td>0.005</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>0.007</td>
<td>0.0048</td>
<td>0.05</td>
<td>0.05</td>
<td>bal</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>R200 58</td>
<td>0.0126</td>
<td>5.30</td>
<td>--</td>
<td>2.72</td>
<td>0.018</td>
<td>0.0083</td>
<td>0.004</td>
<td>0.20</td>
<td>bal</td>
</tr>
<tr>
<td>Ti-6Al-4V*</td>
<td>TE01</td>
<td>0.013</td>
<td>6.30</td>
<td>4.17</td>
<td>--</td>
<td>--</td>
<td>0.0041</td>
<td>0.19</td>
<td>0.19</td>
<td>bal</td>
</tr>
</tbody>
</table>

* Composition reported is average of tested chemistry on top and bottom of forged plate.
Figure 3.1: Optical image of CP-Ti after etching with 0.5% HF.

Figure 3.2: Optical image of Ti-5Al-2.5Sn after etching with 0.5% HF.
Figure 3.3: Equiaxed α lamellar β bi-modal Ti-6Al-4V microstructure (Cortez et al., 2000).

Figure 3.4: Common slip planes and slip plane directions in HCP titanium (Russo and Seagle, 1994).
Figure 3.5: Shape change in solid cube caused by plastic deformation. (a) Undistorted cube; (b) slipped cube with offsets nb; (c) Twinned cube revealing reorientation within twin. Displacements are proportional to distance from twin plane (Hertzberg, 1983).

Figure 3.6: Common slip twinning planes and slip plane directions in HCP titanium (Russo and Seagle, 1994).
Figure 3.7: Effect of temperature and strain rate on twin number density (Nemat-Nasser et al., 1999).

Figure 3.8: Cyclic stress-strain curves for three specimens of α-Ti single crystals. (Gu et al., 1994). Reproduced from Suresh (1998).
Figure 3.9: (a) Misorientation distribution and (b) orientation map showing twins in a sample deformed to true strain of 0.05 in simple compression (Salem et al., 2003).
CHAPTER IV
EXPERIMENTAL PROCEDURE

4.1 Monotonic and Cyclic Tests

All monotonic and low-cycle fatigue (LCF) tests were conducted on an MTS Model 923.14, 20 kip axial-torsion machine in the Mechanical Properties Research Laboratory (MPRL) at Georgia Tech. The CP-Ti monotonic and LCF test specimens, shown in Figure 4.1, were machined at Cincinnati Testing Labs, Cincinnati, Ohio. The Ti-5Al-2.5Sn monotonic and LCF test specimens, shown in Figure 4.2, were machined at Low Stress Grid, Cincinnati, Ohio. The test conditions for the monotonic and LCF tests are listed in Table 4.1 and 4.2, respectively. An elastic modulus check was done per ASTM E111-9 before conducting the monotonic test. Monotonic tests were conducted in strain control up to approximately 12% strain. After reaching this strain, the test control was switched to displacement (stroke) control until failure by fracture. All specimens fractured in the gage section. LCF tests were conducted in strain control per ASTM E606-92. The force was monitored continuously during the test, and failure was defined when the force dropped by half.

4.2 Fretting Tests

4.2.1 Fretting Apparatus and Equipment

An apparatus for conducting fretting experiments was designed and fabricated (Figure 4.3). The design is based on those used in fretting fatigue tests (Hartigan, 2002;
Mutoh et al., 1989). Modifications were made to this design in order to operate in displacement control and also to shift the natural frequency of the apparatus so that the operation was stable at a frequency of at least 5 Hz or greater.

A SATEC 90 kN servo-hydraulic load frame is used to provide displacement actuation. This equipment is located in the Mechanical Properties Research Laboratory (MPRL) at Georgia Tech. A clip gage (MTS #632.02F-20) was used to control the displacement of the fretting pads. Closed-loop feedback mode with peak-valley compensation (PVC) was used to reliably produce fretting contact conditions ranging from stick/partial slip to gross sliding depending on slip amplitude. Two steel extension arms (Figure 4.4) holding the cylindrical fretting pads were rigidly clamped to a fixture (Figure 4.5) attached to the actuator at the bottom. The cylindrical fretting pad radius is 12.7 mm and is 8 mm wide. The 12.7 mm wide x 5 mm thick specimen threaded at the end is screwed into the grip at the top, nearest the load cell. The details of the fretting specimen and pads are shown in Figure 4.6 and 4.7, respectively. Both the specimen and pads were cleaned with acetone prior to testing. The components used to hold the clip gage were clamped around the top grip and on one of the steel arms. Apparatus components are shown in Figures 4.8 through Figure 4.11.

In fretting tests, the controlled variables are the displacement, applied normal force, and choice of contact geometry. An apparatus, ideally with very high stiffness, is designed to rigidly hold the fretting pads. The specimen is attached to an actuator, which supplies the displacement and fretting action. A device such as a clip gage or capacitance gage is placed as close as possible to the fretting pad/specimen interface to continuously measure the displacement of the pad relative to the specimen. Closed loop feedback of
the control system enables the user to control the displacement of the actuator via the clip gage or capacitance gage measurement. The overall goal in creating a fretting apparatus is to make the component holding the pads as rigid as possible to minimize the effect of excessive compliance on the fretting contact. A stiff apparatus is also desired so that tests can be conducted at higher frequencies, which help decrease the time needed to conduct a series of fretting tests. The range of slip amplitudes, number of cycles, and test frequency used in the current work were chosen to enable comparison with the work by Glaeser and Lawless (2001) and Sauger et al. (2000). Sauger et al. (2000) used spherical pads with an applied normal force ranging from 10-1000N with slip amplitudes ranging from 20 μm to 150 μm at frequencies of either 1 Hz or 5 Hz. Glaeser and Lawless (2001) conducted fretting tests at a higher frequency (1000 Hz), using flat pads and slip amplitudes ranging from 20 μm to 75 μm. This range of slip amplitudes was selected from an assessment of representative titanium disk-blade attachments (Glaeser and Lawless, 2001).

The Ti-6Al-4V and Ti-5Al-2.5Sn specimens were machined by Low Stress Grind and polished to a 600 grit finish with the residual polishing marks oriented along the length of the specimen. CP-Ti specimens were machined by Cincinnati Testing Labs and electropolished to reduce the likelihood of twinning in near surface grains due to machining and polishing. The fretting pads were electrical discharge machined (EDM) from the pedigreed Ti-6Al-4V plate by the Woodruff School of Mechanical Engineering machine shop. The pads were not polished in order to avoid modification of the pad radius, rounding at the sides of the pads and also to maintain uniformity in the finish for all of the tests. A Zygo New View 200 light interferometer interfaced with MetroPro software (Zygo Corporation, Middlefield, CT) was used to determine the pad roughness.
for a representative number of pads. An example is shown in Figure 4.12. The root mean square (r.m.s.) value of the pad surface is about 4.28 μm and the Ra is about 3.36 μm. The Ra is the average roughness, which is defined as the integral of the absolute value of the roughness profile height and over the evaluation length. The roughness of the fretting specimen after polishing to a 600 grit finish along the length of the specimen was also measured using a similar procedure. The fretting specimen r.m.s and Ra values are 0.29 μm and 0.17 μm, respectively. These values are used to correct for the effect of roughness on the theoretical half-contact width, α, in order to verify the test conditions, discussed in the next section.

A constant normal force is applied to the pads using a calibrated proving ring, shown in Figure 4.13. This ring was previously used in fretting fatigue tests (Pape, 2002). Contact pressures are achieved in the pad contact area by tightening a load adjusting screw. Four strain gages ( Vishay Measurements EA-96-250AE-350) are bonded to the ring and wired to create a Wheatstone bridge circuit to measure the strain induced by ring loading. The signal from the strain gages was amplified by an Analog Devices 2B30/2B31 signal conditioner. The proving ring strain gage analog signal (in voltage) is converted to a digital signal (in microstrain) by the A/D board in the MTS computer. Throughout each test, an MTS Teststar II system was used to continuously acquire data including time, axial force (frictional force), clip gage displacement, actuator displacement, number of cycles, and normal force.

The proving ring was calibrated by hanging it in a creep frame (Pape, 2002). Creep weights in 10 lb increments were added to a platform attached to the bottom of the ring and the strain gage output was recorded. A linear curve fit of the force versus strain
gage output (in voltage) is used to determine the correct loading for a particular fretting test. The original relationship between voltage and force for this calibration is as follows:

\[ V - V_0 = (0.01)(F) + 0.0014 \]  

(4.1)

where \( V \) is the voltage reading at a given normal force, \( F \), and \( V_0 \) is the initial voltage. A more convenient method can be used by determining the relationship between the change (\( \Delta \)) in microstrain, (\( \mu \epsilon \)), reading obtained using the MTS Teststar II's data acquisition computer system and the applied normal force. The relationship for this calibration is as follows:

\[ F = (0.0918)(\Delta \mu \epsilon) - 2.3241 \]  

(4.2)

Tests 1 through 11 were completed with this calibration, after which some grounding problems were encountered and loose wires on the ring had to be resoldered. This necessitated a new ring calibration that was completed using a calibrated load cell ((Transducer Techniques #DPM-3) inserted between the loading screw and the fretting pads after the apparatus was assembled in the servo-hydraulic machine. The calibration of the load cell was verified by placing it between platens in the servo-hydraulic machine used in the fretting tests, then slowly applying a force in 10 lb increments. The load cell reading was recorded along with the applied force. The calibration equation for Tests 12-25 is as follows:

\[ F = (0.0732)(\Delta \mu \epsilon) - 3.5021 \]  

(4.3)

Fretting tests were conducted at 5 Hz. The peak-valley compensation feature of MTS Teststar II's control system was used to reach the desired displacement limits for this loading frequency. The parameters used are given in Table 4.3.
4.2.2 Experimental Program

The theoretical size of the contact area and pressure distribution between the pad and specimen were obtained using Hertzian contact theory. A summary of the Hertz contact theory is given by Johnson (1985). Only the pertinent results are summarized here.

For the 2-D problem of contact between cylinders, the contact half-width, $a$, is given by

$$a^2 = \frac{4FR}{\pi E^*}$$  \hspace{1cm} (4.4)

where $F$ is the normal force, $t$ is the pad width, and $R$ is the combined radius of curvature for each body, given by

$$\frac{1}{R} = \frac{1}{R_1} + \frac{1}{R_2}$$  \hspace{1cm} (4.5)

For a cylinder of radius $R_1$ in contact with a flat surface, $R_2$ goes to infinity.

$E^*$ is the effective elastic constant defined for an isotropic body,

$$\frac{1}{E^*} = \frac{1-v_1^2}{E_1} + \frac{1-v_2^2}{E_2}$$  \hspace{1cm} (4.6)

where $v$ is Poisson's ratio.

The equations above are based on the assumption that contact occurs at all points throughout the nominal contact area, which is only true for an ideally smooth surface.

The fretting pads used in the experiments were not polished in order to avoid inadvertent modification of the pad radius or edge rounding, and so cannot be considered smooth. The real contact pressure of a rough surface at discrete spots (i.e., asperity contacts) are much higher than the pressure predicted by Hertz theory when it is applied to the whole
contact (Johnson, 1982). Therefore, the true contact over an area of a rough surface is smaller than the apparent area of contact.

Greenwood and Williamson (1966), Greenwood and Tripp (1967) and Johnson (1975, 1982) have studied the effect of roughness on contact area for a sphere in contact with a rough flat surface. Correction factors have been developed to partially account for the surface roughness. The deformation of the contact between a smooth sphere with a nominally flat random rough surface is primarily dependent upon the non-dimensional parameter:

$$\alpha = \frac{\sigma}{\delta_s} = \frac{\sigma R}{a_o^2} = \frac{\left(16RE^{\nu^2}\right)^{1/3}}{9F^{1/3}}$$

(4.7)

where $\sigma$ is the standard deviation of the surface roughness, $\delta_s$ and $a_o$ are the Hertzian (i.e., smooth surface) bulk normal compressive displacement and nominal contact radius, respectively, for a given applied force $F$. For practical calculations, $\sigma$ can be taken to be the combined (r.m.s.) value of the roughness of both surfaces. For a very rough surface ($\sigma=1.85$), the actual contact radius can be as high as 3.0 times the nominal Hertzian solution for a smoother surface ($\alpha = 0.047$), as illustrated in Figure 4.14.

Some difficulties arise when attempting to modify this solution for use with cylindrical contact geometry because of the constraints on the bulk compressive displacement term $d$ for 3-D vs. 2-D contact solutions. The elastic contact of rough cylinders has been derived by Lo (1969). Unfortunately, microscopic parameters such as the asperity density and asperity radius must be known to obtain an exact solution. Therefore, only the general trend in contact width changes due to roughness of two cylinders in contact will be shown here. For smooth contact between cylinders, the half-
contact width, \( a \), was given earlier (Equation 4.4). The nominal Hertzian half-contact width is proportional to the square root of the normal force, \( F \). In other words,

\[
a \propto F^{m}
\]  
(4.8)

where \( m = 0.5 \). Lo (1969) determined that when the contacting surfaces are rough, \( m \) ranges between 0.055 for a high load and 0.30 for a low load. Note that \( a \) is a number that is much less than 1 for the loading conditions used in the current work, so the smaller exponent (i.e. \( m=0.055 \)) results in a larger half-contact width than when \( m=0.3 \). This translates to a rough surface half-contact width (all other variables held constant) between 3.4 times and 15 times the nominal Hertzian half-contact width.

Even though the pads used in the current study are cylindrical instead of spherical, Equation 4.7, may be used as a rough estimate. For a combined Ti-6Al-4V pad and Ti-6Al-4V specimen r.m.s. equal to 4.57, and using a normal force equal to 703 N, the non-dimensional parameter, \( \alpha \), is 2.76. Therefore, the scar width observed after fretting tests should be approximately equal to three or more times the theoretical smooth contact width plus the applied slip range.

In order to determine the proper normal pad force for a given material, the pressure required to initiate yielding according to the von Mises criterion must be calculated. From Hertzian contact theory (Johnson, 1985), the pressure distribution on the surface is represented by \( p(x) \), which goes to zero outside the contact area.

\[
p(x) = \frac{2F}{\pi a^2} \sqrt{a^2 - x^2} \quad \text{for} \quad |x| \leq a
\]  
(4.9)

\[
p(x) = 0 \quad \text{for} \quad |x| > a
\]  
(4.10)

The maximum pressure at the contact, \( p_m \), occurs at \( x = 0 \),
\[ P_0 = \frac{2F}{\pi a} = \sqrt{\frac{FE}{\pi R}} \]  

(4.11)

When friction is zero, yielding first occurs at a location along the z-axis (see Figure 4.15 (a)). The force required to initiate yielding, \( F_y \), can be determined by plotting \( J_2/(\sigma_y)^2 \) versus the normalized depth \((z/a)_n\), where \( z \) is the depth below the surface and \( a \) is the contact half-width given by Equation (4.4). An example of this plot is shown in Figure 4.15 (b) for Ti-6Al-4V for \( R = 12.7 \text{ mm} \).

The stress field under the contact surface depends on \( P_0 \) and \( a \). In particular, the principal stress distributions along the z-axis (where \( x = 0 \)) are as follows (Johnson, 1985):

\[
\sigma_1 = -P_0 \left[ 1 + \frac{2(z/a)^2}{1 + (z/a)^2} \right]^{1/2} - \frac{2(z/a)}{1 + (z/a)^2},
\]

\[
\sigma_2 = -P_0 \left[ 1 + \frac{(z/a)^2}{1 + (z/a)^2} \right]^{1/2}, \text{ and}
\]

\[
\sigma_3 = \nu(\sigma_1 + \sigma_2) \text{ for plane strain}
\]

(4.12)

where \( \sigma_1, \sigma_2, \) and \( \sigma_3 \) are the principal stresses. The von Mises yield criterion is given by

\[
J_2 = \frac{1}{6} \left( (\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 \right) = k^2 = \frac{\sigma_y^2}{3}
\]

(4.13)

where \( J_2 \) is the second invariant of the deviatoric stress tensor, and \( k \) and \( \sigma_y \) represent the yield stress of the material in simple shear and simple tension (or compression), respectively.

The peak in the curve indicates the depth where yield initiates, which occurs when \( J_2 \) is \( 0.098P_0^2 \) at a depth of \( 0.74a \) for Ti-6Al-4V. The critical value of \( P_0 \) according to the von Mises criterion can then be determined using Equation 4.13.
Substitution of this value into Equation 4.11 gives the force \( F_y \) required to initiate yielding in Ti-6Al-4V for the given pad geometry. Note that these relations depend on the Poisson’s ratio, so the coefficients in Equation 4.14 vary with material. Results for each material are given in Table 4.4.

The fretting experimental program is shown in Table 4.5. The loading conditions were chosen to enable comparison with fretting tests conducted by Glaeser and Lawless (2001) and Sauger et al. (2000). The normal load applied to the CP-Ti specimens is the minimum force that can be safely applied to the pads using the proving ring. The theoretical contact width \( (2a) \) for a smooth surface according to Hertz contact theory is added to the applied slip amplitude to determine the total theoretical contact width for each test.

For each series of tests, a constant normal force, \( F \), was applied to the pads at a certain percentage of the force \( (P/P_s) \) required to produce yielding according to the Von Mises criterion (Johnson, 1985). The same procedure was used for CP-Ti and Ti-5Al-2.5Sn. The slip amplitude was modified to produce a contact condition of either gross sliding or partial slip/stick. All tests were conducted at a frequency equal to 5 Hz.

4.3 Metallography Procedure

Meticulous specimen preparation was required for this work because diffraction patterns obtained using EBSD are extremely sensitive to very small variations in surface topography. The top 10-50 nm of the specimen must be representative of the region in which the diffraction pattern is obtained (Randle and Engler, 2000). Because the
specimen is highly tilted for the analysis, the diffraction zone is very shallow. This means that the microstructure of the specimen must not be obscured in any way by mechanical damage (i.e., grinding/polishing), surface layers (i.e., oxidation, coatings), or contamination. Even more importantly, data interpretation of a poorly prepared specimen can be misleading. The diffuseness or absence of an EBSD pattern is some measure of the amount of plastic strain in the lattice, or may indicate that the grain size is smaller than the probe size (Tucker et al., 2000). However, these interpretations are only valid after correct specimen preparation.

The specimen was prepared in a number of steps: sectioning, mounting, grinding, diamond polish, and a final polish. In every step, the goal was to minimize the residual strain in the specimen as much as possible. The scar was sectioned in half in the longitudinal direction at slow speed using a LECO® VC-50 precision cutter and a Buehler 15 HC diamond saw lubricated continuously with LECO® VC-50 cutting oil. One half of the section was reserved for EBSD studies. The other half of the section was reserved for subsurface scanning electron microscope (SEM) observations, which required etching with 0.5% hydrofluoric acid. These specimens were subsequently repolished and used for nanoindentation tests.

The Ti-6Al-4V and Ti-5Al-2.5Sn specimens were mounted in Buehler Epo-Quick epoxy with a conductive filler, then ground and polished. Because CP-Ti is so soft, mechanical polishing induces near surface residual strains that make indexing of EBSD patterns difficult or impossible if mechanical polishing alone is performed. The CP-Ti specimens used for EBSD was electropolished after grinding and mechanical polishing.
using the same procedure as Ti-6Al-4V and Ti-5Al-2.5Sn. The electropolishing procedure used for CP-Ti will be discussed shortly.

Grinding is done by hand using a tap water cooled grinding wheel (Buehler Ecomet®3) with Struers P320, 500, 800, and 1200 grit SiC papers for approximately 10 minutes at 150 revolutions per minute for each paper. It is critical that the marks left by the previous paper be removed with every subsequent paper. To insure this and also to help keep the specimen flat, an arrow is marked on the back of the specimen so that the sanding direction can be rotated 90 degrees with every change in paper.

The intermediate polishing step is completed using polycrystalline diamond spray (Struers DP-Spray) in steps of 9, 6, 3, and 1 μm using various Struers cloths. All metallographic supplies are from Struers, except where noted. For this process, diamond paste was determined to be either to inconsistent in diamond size (i.e., quality) or too difficult to apply and achieve repeatable results. Final polishing was done using a 0.02 μm colloidal silica solution (Buehler Mastermet 2). All polishing steps were completed using an automatic polisher (Struers RotoPol15) after which the specimens were cleaned with distilled water. Optimum selection of lubricant and cloth for each step minimizes the time and pressures required to remove previous scratches. The combination of polishing agent, cloth, and lubricant shown in Table 4.6 have produced results that are consistent and repeatable for all materials. However, the pressures and times are approximate. Before proceeding with the next step, it is necessary to place the specimen in an optical microscope, ideally equipped with a cross-polarized light filter, to verify that previous scratches have been removed. An additional electropolishing step was performed on CP-Ti specimens used for EBSD.
Note that this lengthy procedure is only required for EBSD analysis specimens in order to limit very near surface residual strains that result from traditional mechanical polishing. The number of grinding and polishing steps may be reduced for specimens that are used for other procedures, such as SEM or nanoindentation.

The CP-Ti specimens had to be mounted in non-conductive diallyl phthalate to facilitate electropolishing. Unfortunately, a non-conductive mount is not suitable for EBSD studies. In some cases, a light carbon coating can be applied to reduce charging. However, this procedure was tried on Ti-6Al-4V specimens, and even a thin carbon coating was enough to make indexing patterns near the fretted surface difficult. Therefore, the decision was made to remove the CP-Ti specimen from the mount after electropolishing was finished. Also, titanium is ideally electropolished in a perchloric acid solution. Attempts made to have CP-Ti electropolished using other chemical solutions proved unsuitable for EBSD work due to excessive etching. Since perchloric acid can be extremely dangerous and unstable, it requires very special handling and equipment. The Air Force Research Laboratory at Wright-Patterson Air Force Base in Dayton, Ohio happened to be one of the few locations that have the facilities to electropolish using perchloric acid, so the specimens were shipped there after final polishing with colloidal silica. After much trial and error, a suitable electropolishing procedure was finalized. Electropolishing is done at -50°C, with moderate agitation. The specimen is polished face up in a cylindrical cathode for about 8 minutes, in voltage control mode at 25 volts. The electropolishing recipe for CP-Ti is given below:

- Methanol 590 ml
- Butyl cellulose 350 ml
• Perchloric acid (60% concentration) 60 ml

Again, perchloric acid can be very dangerous, therefore, it is highly recommended that electropolishing only be done by an experienced professional.

4.4 Microstructural Characterization and Surface Analysis

4.4.1 Scanning Electron Microscopy

Scanning electron microscopy (SEM) using a secondary electron signal was used to visually observe subsurface microstructural features that are too small to be clearly seen in an optical microscope. Secondary electrons are produced as a result of the inelastic scattering between the incident electron beam and the sample. Secondary electrons are low energy, and are only able to escape from the near surface of the sample. The spread of the incident beam is minimal, therefore, images with high spatial resolution of the order of the incident beam diameter can be acquired. Changes in signal level due to secondary electron emission can arise from changes in both topography and composition of the material, so the accelerating voltage (also referred to as kV) and beam current for a given image need to be optimized. In general, a higher kV provides more contrast between features with different composition, but results in greater charging. A lower kV usually provides better image resolution of topographical features, but contrast is not as good. The optimal accelerating voltage to obtain images in Ti-6Al-4V, Ti-5Al-2.5Sn and CP-Ti appears to be about 10kV.

All specimens used for SEM observations were etched by swabbing for 30 seconds with hydrofluoric acid (HF) diluted to 0.5% with distilled water. This substance can be very dangerous; therefore, special handling and equipment is required. The
cleanroom facilities at MiRC at Georgia Tech were used for etching, as HF is a common agent used for treatment of silicon wafers. Even with etching, however, the topographical features of CP-Ti and Ti-6Al-2.5Sn are not observed as easily as with Ti-6Al-4V due to the fact that they are mostly single phase materials.

A LEO Gemini Model 1730 thermally assisted field emission gun (FEG) SEM was used in the subsurface analysis. An FEG SEM has much better resolution than a tungsten filament SEM, and so is preferred for observing features at magnification greater than about 2000-3000X.

4.4.2 Electron Backscatter Diffraction (EBSD)

The EBSD system at Georgia Institute of Technology is an attachment to the LEO Gemini FEG SEM. The system hardware includes a retractable Hamamatsu C2400 low-light silicon intensified target (SIT) camera for Kikuchi pattern acquisition with a phosphor screen. The EBSD analysis is set up within Channel 5 Flamenco software (HKL Technology, 2000) which automatically indexes the Kikuchi patterns obtained by the EBSD hardware.

A number of factors had to be considered before setting up an EBSD experiment. The estimated depth of damage, width of the fretting scar, initial grain size, and surface finish, among other factors, affect how an EBSD experiment will be performed. A higher accelerating voltage tends to increase the intensity of the Kikuchi patterns, but decreases the resolution. Also, in mounted specimens, a higher accelerating voltage increases the amount of charging, which results in beam drift that could be significant over the duration of time required to perform an EBSD test.

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The depth of damage in a fretted specimen is not known *a priori*. However, a reasonable estimate can be made based on prior observations by SEM. The sampling depth for a given material was selected to be at least one grain deep, with the maximum grain diameter in the virgin material chosen as the minimum sample depth.

Other factors considered in choosing the EBSD sample depth are as follows:

- Tendency of near surface material to undergo changes in mechanical properties (i.e. hardness) is estimated to be on the order of 1-3 grain diameters (Hirth and Rigney, 1979).

- Grain refinement or coarsening that may occur due to fretting loading can only be determined if the sample area is large enough to encompass the largest grain size as determined from the as-received material.

The maximum grain diameter, rather than the average grain diameter, was used to compare results between tests because the average grain diameter measurement appeared to be very dependent upon the percent indexation of a particular EBSD test and also upon how the noise reduction algorithm was performed. For the current study, a grain boundary is defined as having a misorientation of 10° or greater. The maximum grain diameter calculation was relatively insensitive to differences between EBSD tests, and so is believed to be a better variable to use for comparison purposes. Also, the number of sampled grains used in the texture analysis is usually greater than 100, so it is not likely that one large grain would disproportionately skew the results. A table that lists the test parameters for each EBSD experiment is given in Appendix C.
4.4.3 Nanoindentation Technique

Local material properties such as hardness and elastic modulus of the region directly beneath the fretting region were determined using instrumented indentation testing via an MTS NanoIndenter® XP and MTS software Version 4.06A. This method was developed to determine mechanical properties even when the indentations are too small to be easily imaged. Instead of imaging, the contact area is estimated using analytical functions established by analyzing the load-displacement data for an indentation (MTS, 2001). A thorough treatment of nanoindentation fundamentals is covered by Oliver and Pharr (1992).

At any time during loading, the total displacement, \( h \), shown in Figure 4.16 is written as:

\[
h = h_i + h_f \tag{4.15}
\]

where \( h_i \) is the vertical contact distance (a.k.a. the contact depth), \( h_i \) is the surface displacement at the contact perimeter, and \( h_f \) is the final displacement after complete unloading [Oliver and Pharr, 1992, MTS, 2001]. Both elastic and plastic deformation cause a hardness impression to be formed that conforms to the shape of the indenter to the contact depth, \( h_i \). Only the elastic portion of the displacement is recovered when the indenter is withdrawn, which allows for the determination of the elastic properties of a material.

Data are commonly obtained from one complete cycle of loading and unloading, as shown in Figure 4.17. The load-displacement data acquired during the unloading portion is fit to the power-law relation:

\[
P = B(h - h_f)^n \tag{4.16}
\]
where $P$ is the load applied to the test surface, $h$ is the resulting penetration, and $B$ and $m$ are empirically determined fitting parameters. This relation does not always provide an adequate description of the entire unloading curve, especially for thin films, therefore, the contact stiffness, $S$, is determined by fitting the upper 25% to 50% of the unloading data.

The unloading stiffness, $S$, is established by differentiating Equation (4.16) and evaluating at the maximum depth of penetration, $h = h_{\text{max}}$, i.e.,

$$S = Bm(h_{\text{max}} - h)^{m-1} \quad (4.17)$$

The reduced modulus, $E_r$, is related to the contact stiffness, $S$, and contact area, $A$ by the equation:

$$E_r = \frac{\sqrt{\pi S}}{2\beta \sqrt{A}} \quad (4.18)$$

where $\beta$ is a constant that depends on the geometry of the indenter. For axisymmetric indenter geometries, the elastic modulus of the test material, $E$, is then calculated using the expression:

$$\frac{1}{E_r} = \left(1 - \nu^2\right) \left(1 - \nu_i^2\right) \frac{1}{E} \quad (4.19)$$

where $\nu$ is the Poisson’s ratio for the test material, and $E_i$ and $\nu_i$ are the elastic modulus and Poisson’s ratio of the indenter, respectively. Because this equation was derived for axisymmetric indenters, it technically applies only to circular contacts and $\beta = 1$.

However, this equation can be used for non-axisymmetric indenters when empirically determined $\beta$ values are included in Equation (4.18). A Berkovich indenter ($\beta = 1.034$) with an angle of $65.3^\circ$ between the tip axis and the faces of the triangular pyramid was used for the analysis.

The contact depth, $h_c$, is generally different from the total penetration depth and is estimated by the relation:
where \( c \) is a constant which depends on indenter geometry. For a Berkovich indenter, \( c = 0.75 \). Equation 4.20 is based on elastic theory, but has been shown to work well when the contact caused significant plastic deformation. However, it cannot account for the plastic phenomenon of pile-up.

Finally, the projected contact area, \( A \), is calculated by evaluating an empirically determined area function at the contact depth \( (h_c) \).

\[
A = f(h_c) \tag{4.21}
\]

The method described above requires no imaging be done. This method is based on the assumption that the elastic modulus is independent of indentation depth. Justification for this assumption is presented in Oliver and Pharr (1992). A first estimate of the area function is based on large indentations made in aluminum, and assumes perfect Berkovich indenter geometry.

\[
A(h_c) = 24.5h_c^2 \tag{4.22}
\]

In reality, the tip of the indenter is blunted, with a tip radius on the order of 50 nm. An initial guess at the area function is made by fitting the \( A \) vs \( h_c \) data to the relationship

\[
A(h_c) = 24.5h_c^2 + C_1h_c^1 + C_1h_c^{1/2} + C_2h_c^{1/4} + \ldots + C_8h_c^{1/128} \tag{4.23}
\]

where \( C_1 \) through \( C_8 \) are constants. All terms except the first describe deviations from the perfect Berkovich geometry due to blunting at the tip. Constants \( C_1 \) through \( C_8 \) are established for each individual indenter and provided by the indenter manufacturer. The procedure to determine the area is iterated several times until convergence is achieved.

The hardness of the test surface is then determined using the equation,
where $P$ is the contact load and $A$ is the projected contact area for a given load.

A nanoindentation experiment can be conducted in basically one of two ways: load control or displacement control. Most are conducted in load control. However, there are some advantages to using displacement control. With the software that is currently available, one can obtain the hardness and elastic modulus as a continuous function of depth from a single indentation experiment using a “continuous stiffness measurement” (CSM). This method allows precise control of the penetration depth, which is most useful when testing very thin films and coatings (Li and Bhushan, 2002). As the name implies, the stiffness is measured continuously throughout a test, unlike load controlled tests that measure properties upon unloading.

The CSM technique is accomplished by imposing a small sinusoidally varying signal on top of a DC signal that drives the motion of the indenter (Li and Bhushan, 2002). The response of the system is analyzed by means of a frequency specific amplifier, allowing the measurement of contact stiffness at any point along the loading curve, not just at the point of unloading. The measurements can be made at very small penetration depths and it also enables measurement of materials that have properties that vary with depth, such as non-homogeneous, polymeric, or multi-layered structures.

An array of indents spaced between 15 – 30 $\mu$m was used to cover the region of interest. A Berkovich indenter was used for this study because it produces plasticity at very low loads, it has good manufactured quality, and it minimizes the influence of friction (MTS, 2001). It is recommended that successive indentions using a Berkovich indenter should be separated by at least 20 to 30 times the maximum penetration depth in
order to avoid interference. The degree of required surface smoothness depends on the magnitude of the measured displacements and the tolerance for uncertainty for a given contact area [MTS, 2001]. For this study, all specimens received a final polish of 0.02 μm colloidal silica.

4.4.4 Energy Dispersive X-ray Analysis (EDX)

Energy dispersive X-ray (EDX) analysis was conducted in order to more easily distinguish the regions in which substantial oxidation had occurred near the fretted surface. The emission of an X-ray photon has a characteristic energy that is specific to the element. Therefore, detection of these X-rays gives information about the elemental composition of the sample, both in terms of quantity and distribution (Oxford Instruments, Oxon, England).

The concentration is represented as "counts" along a line in the ISIS mapping function. The counts correspond to the value of the incident electron beam energy, and so is a measure of the intensity of the signal corresponding to a particular element. The counts of each element are then integrated in the spectrum analysis to determine the total atomic or weight percent of each element. The EDX detector and spectrum analysis software are an Oxford Instruments (Oxon, England) model 7426 and Inca Version 3.04 (Oxford Instruments, Oxon, England), respectively. This equipment is an attachment to the LEO Gemini FEG SEM.

All EDX analysis was conducted using secondary electrons at an accelerating voltage of 10kV to enable detection of lighter elements such as oxygen. Oxygen was not detected when a higher accelerating voltage of 30kV was used.
### Table 4.1: Monotonic test matrix.

<table>
<thead>
<tr>
<th>Material</th>
<th>Specimen</th>
<th>Strain rate ($\text{s}^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-Ti</td>
<td>CP-1</td>
<td>5e-3</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>CP-2</td>
<td>5e-5</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>LT5-2</td>
<td>5e-3</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>LT5-1</td>
<td>5e-5</td>
</tr>
</tbody>
</table>

### Table 4.2: LCF test matrix.

<table>
<thead>
<tr>
<th>Material</th>
<th>Specimen</th>
<th>Strain range (%)</th>
<th>Strain rate ($\text{s}^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-Ti</td>
<td>AR-L211</td>
<td>0.5</td>
<td>5e-3</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>AR-L121</td>
<td>1.0</td>
<td>5e-3</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>AR-L311</td>
<td>1.6</td>
<td>5e-3</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>AR-L221</td>
<td>2.75</td>
<td>5e-3</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>LT5-3</td>
<td>1.0</td>
<td>5e-3</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>LT5-4</td>
<td>1.5</td>
<td>5e-3</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>LT5-6</td>
<td>2.0</td>
<td>5e-3</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>LT5-5</td>
<td>2.75</td>
<td>5e-3</td>
</tr>
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</table>

### Table 4.3: Peak valley compensation calibration constants

<table>
<thead>
<tr>
<th>P gain</th>
<th>I gain</th>
<th>Convergence rate (%)</th>
<th>Sensitivity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>17.8</td>
<td>3.00</td>
<td></td>
<td>0.1</td>
</tr>
</tbody>
</table>

### Table 4.4: Load, normalized depth, and force per unit length required to initiate yielding according to von Mises criterion.

<table>
<thead>
<tr>
<th>Constant</th>
<th>Ti-6Al-4V</th>
<th>CP-Ti</th>
<th>Ti-5Al-2.5Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma_y$ (MPa)</td>
<td>930</td>
<td>185</td>
<td>901</td>
</tr>
<tr>
<td>E (GPa)</td>
<td>118</td>
<td>105</td>
<td>122</td>
</tr>
<tr>
<td>Poisson's ratio, $\nu$</td>
<td>0.349</td>
<td>0.34</td>
<td>0.33</td>
</tr>
<tr>
<td>Pad radius, R (mm)</td>
<td>12.7</td>
<td>12.7</td>
<td>12.7</td>
</tr>
<tr>
<td>Pad thickness, t (mm)</td>
<td>8</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>$(p_0), (MPa)$</td>
<td>1720</td>
<td>340</td>
<td>1648</td>
</tr>
<tr>
<td>$l_{0}/r_0$ at yield</td>
<td>0.74</td>
<td>0.73</td>
<td>0.73</td>
</tr>
<tr>
<td>$F_y$ (MPa/mm)</td>
<td>1758</td>
<td>74.4</td>
<td>1600</td>
</tr>
</tbody>
</table>

88
<table>
<thead>
<tr>
<th>Test #</th>
<th>Specimen material</th>
<th>Specimen number</th>
<th>Fp0(N)</th>
<th>F/Fy</th>
<th>Nominal slip amp (µm)</th>
<th>Cycles</th>
<th>2a + ref slip range (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>113-S612</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>410.9</td>
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</tr>
<tr>
<td>2</td>
<td>113-S612</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>410.9</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>113-S612</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>410.9</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>113-S612</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>530.9</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>113-S612</td>
<td>703</td>
<td>0.05</td>
<td>30</td>
<td>100000</td>
<td>350.9</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Ti-6Al-4V</td>
<td>703</td>
<td>0.05</td>
<td>15</td>
<td>100000</td>
<td>300.9</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>113-S612</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>410.9</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>dummy</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>410.9</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>113_S612_2</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>410.9</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>113_S612_2</td>
<td>703</td>
<td>0.05</td>
<td>60</td>
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<td>410.9</td>
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<td>11</td>
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<tr>
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<td>703</td>
<td>0.05</td>
<td>120</td>
<td>200</td>
<td>530.9</td>
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<tr>
<td>21</td>
<td>CRP-Ti</td>
<td>294</td>
<td>0.5</td>
<td>60</td>
<td>100000</td>
<td>314.0</td>
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<tr>
<td>22</td>
<td>AR-S131</td>
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<td>0.5</td>
<td>60</td>
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<td>23</td>
<td>AR-S131</td>
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<td>100000</td>
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<td>30</td>
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<tr>
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<td>AR-S131</td>
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<td>0.5</td>
<td>15</td>
<td>100000</td>
<td>224.0</td>
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<tr>
<td>12</td>
<td>STS_1</td>
<td>640</td>
<td>0.05</td>
<td>120</td>
<td>100000</td>
<td>516.2</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>STS_1</td>
<td>640</td>
<td>0.05</td>
<td>1/c0</td>
<td>100000</td>
<td>516.2</td>
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<tr>
<td>14</td>
<td>STS_1</td>
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<td>100000</td>
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</tr>
<tr>
<td>15</td>
<td>STS_1</td>
<td>640</td>
<td>0.05</td>
<td>30</td>
<td>100000</td>
<td>336.2</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>Ti-6Al-2.5Sn</td>
<td>640</td>
<td>0.05</td>
<td>15</td>
<td>100000</td>
<td>306.2</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>STS_1</td>
<td>640</td>
<td>0.05</td>
<td>120</td>
<td>100000</td>
<td>516.2</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>STS_1</td>
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<td>0.05</td>
<td>60</td>
<td>100000</td>
<td>396.2</td>
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</tr>
<tr>
<td>19</td>
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<td>640</td>
<td>0.05</td>
<td>120</td>
<td>200</td>
<td>516.2</td>
<td></td>
</tr>
</tbody>
</table>
Table 4.6: Metallographic preparation procedure.

<table>
<thead>
<tr>
<th>Agent</th>
<th>Cloth</th>
<th>Lubricant</th>
<th>Speed (rpm)</th>
<th>Pressure</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>sandpaper</td>
<td>P320 SiC</td>
<td>Tap water</td>
<td>150</td>
<td>hand</td>
<td>As required</td>
</tr>
<tr>
<td>sandpaper</td>
<td>P500 SiC</td>
<td>Tap water</td>
<td>150</td>
<td>hand</td>
<td>As required</td>
</tr>
<tr>
<td>sandpaper</td>
<td>P800 SiC</td>
<td>Tap water</td>
<td>150</td>
<td>hand</td>
<td>As required</td>
</tr>
<tr>
<td>Diamond spray 9 μm</td>
<td>MD-Pan</td>
<td>DP- Blue (alcohol based)</td>
<td>150</td>
<td>20 N</td>
<td>2 min</td>
</tr>
<tr>
<td>Diamond spray 6 μm</td>
<td>MD-Mol</td>
<td>DP- Blue</td>
<td>150</td>
<td>20 N</td>
<td>2 min</td>
</tr>
<tr>
<td>Diamond spray 3 μm</td>
<td>MD-Mol</td>
<td>DP- Red (oil based)</td>
<td>150</td>
<td>20 N</td>
<td>2-4 min</td>
</tr>
<tr>
<td>Diamond spray 1 μm</td>
<td>MD-Nap</td>
<td>DP – Blue</td>
<td>150</td>
<td>20 N</td>
<td>2-4 min</td>
</tr>
<tr>
<td>Colloidal silica 0.02 μm</td>
<td>MD-Chem</td>
<td>Distilled water</td>
<td>150</td>
<td>10 N</td>
<td>10 min or more</td>
</tr>
</tbody>
</table>
Figure 4.1: Monotonic and LCF test specimen design used for CP-Ti.

Figure 4.2: Monotonic and LCF test specimen design used for Ti-5Al-2.5Sn.
Figure 4.3: Fretting apparatus used in fretting tests.
Figure 4.4: Extension arm.
Figure 4.5: Lower clamp design.
Figure 4.6: Specimen design used for fretting tests (a) Ti-6Al-4V and Ti-5Al-2.5Sn (b) CP-Ti.
Figure 4.7: Fretting pad design.
Figure 4.8: Upper clamp design.
Figure 4.9: Apparatus components (a) extension arm bracket and (b) loading ball piston.
Figure 4.10: Lower clip gage bracket.
Figure 4.11: Upper clip gage bracket.
Figure 4.12: Representative pad roughness measurement.
Figure 4.13: Proving ring (Pape, 2002).
Figure 4.14: Contact of a smooth sphere with a nominally flat (a) very rough surface, α = 1.85, (b) slightly rough surface, α = 0.047 (Johnson, 1982).

Figure 4.15: (a) Coordinate system and (b) normalized effective stress along z-axis for Ti-6Al-4V.
Figure 4.16: Schematic of indentation showing parameters used in indentation analysis (Oliver and Pharr, 1992).

Figure 4.17: Loading versus displacement curve for a typical nanoindentation test (Oliver and Pharr, 1992).
CHAPTER V
RESULTS AND DISCUSSION

5.1 Mechanical Properties

5.1.1 Monotonic and Cyclic Stress-Strain Response

Monotonic and low cycle fatigue (LCF) tests were conducted at Georgia Tech on CP-Ti and Ti-5Al-2.5Sn to obtain most of the elastic, plastic, and fatigue constants listed in Table 5.1, 5.2, and 5.3, respectively. Most of the Ti-6Al-4V constants were obtained from the Air Force High Cycle Fatigue (HCF) program investigation (Gallagher, 2000; Eylon, 1998; Duryak, 1999). The monotonic and cyclic stress-strain curves for Ti-6Al-4V were extracted from data and/or test plots supplied by Duryak (1999) shown in Figure 5.1 and Kurath (1999) shown in Figures 5.2 and 5.3. The strain rate was not reported for any of the tests on Ti-6Al-4V.

A comparison of the monotonic and cyclic stress-strain curves for Ti-6Al-4V, CP-Ti, and Ti-5Al-2.5Sn is shown in Figure 5.4. The bulk material response of both Ti-6Al-4V and Ti-5Al-2.5Sn are similar and summarized below.

- The elastic modulus (i.e., stiffness) is similar.
- Nearly elastic-perfectly plastic.
- Exhibits cyclic softening.

The bulk material response of CP-Ti is different than Ti-6Al-4V or Ti-5Al-2.5Sn, most notably in the general tendency to cyclically harden at a strain amplitude greater
than about 0.5% instead of soften. It also tends to slightly strain harden under monotonic tension. CP-Ti appears to be less strain rate dependent than Ti-5Al-2.5Sn and is less stiff than either Ti-6Al-4V or Ti-5Al-2.5Sn. The cyclic stress-strain curve of CP-Ti indicates a mixed behavior, where it is cyclically stable or softens slightly at strain amplitudes lower than about 0.5%, but strain hardens afterwards. Both CP-Ti and Ti-5Al-2.5Sn exhibit a “dip” at relatively low plastic strains. This phenomenon is linked to the propensity for twin formation in pure titanium. A marked increase in the cyclic hardening rate of α-Ti single crystals coincides with an increase in the occurrence of cyclic twins (Gu et al., 1994). However, Ti-5Al-2.5Sn does not exhibit significant twinning at the temperature and strain rate used in the current study, and so it is less likely that twinning is the primary factor in rationalizing this behavior in Ti-5Al-2.5Sn.

Further details regarding CP-Ti and Ti-5Al-2.5Sn LCF tests conducted at Georgia Institute of Technology are included in Appendix A.

The 0.2% offset yield strength (σ₀) and ultimate tensile strength (σₘ) were determined in monotonic strain-controlled tests at strain rates of 5x 10⁻⁸ s⁻¹ and 5x 10⁻⁹ s⁻¹. The elastic modulus (E) for these two materials were determined from the slope of the stress-strain curve. The Poisson’s ratio (ν) for CP-Ti and Ti-5Al-2.5Sn were obtained from Efunda (2003).

True stress (σ) versus true plastic strain (εₚ) from monotonic tension tests can be expressed as a power function of the Ramgoud-Osgood form (Bannantine et al., 1990):

$$\sigma = H(\varepsilon_p)^n$$

(5.1)

where n is the strain hardening exponent and H is the strength coefficient. Engineering plastic strain values between 0.013% and 16.3% were used to determine these constants.
for Ti-5Al-2.5Sn. For CP-Ti, engineering plastic strain values between 0.22% and 16.7% were used. The flow exponent, m, which is also the inverse strain rate sensitivity used in rate-dependent computational analysis (Goh et al., 2001; Goh, 2002) was estimated by a fit of the plastic strain rate versus the monotonic yield strength for CP-Ti and Ti-5Al-2.5Sn

\[ \dot{\varepsilon}_p = A\sigma^n \]  

(5.2)

5.1.2 Fatigue Properties

The strain-life plots for Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti obtained from stabilized half-life deformation response are shown in Figure 5.5, Figure 5.6, and Figure 5.7, respectively. A comparison of the strain-life curves for all three materials is shown in Figure 5.8. Coincidentally, the total strain-life curves for Ti-5Al-2.5Sn and CP-Ti are very similar. However, the Ti-5Al-2.5Sn strain-life behavior is dominated by elastic strain, while CP-Ti is dominated by plastic strain. The strain-life and stabilized half-life deformation data for Ti-5Al-2.5Sn and CP-Ti are given in Tables 5.4 and 5.5, respectively.

5.2 Fretting Experiments

The fretting contact conditions can be determined by analyzing the frictional force versus displacement hysteresis loops throughout a fretting test. Stabilized frictional force (F) versus clip gage displacement (δ) hysteresis loops for different displacement amplitudes (δs) from fretting tests conducted on Ti-6Al-4V are shown in Figure 5.9.

The total frictional energy expended per cycle is given by the area enclosed within the loop (Hills and Nowell, 1994). As such, the form of these hysteresis plots
indicate whether the contact is in a partial slip/stick, or gross sliding condition. The coefficient of friction, \( \mu \), is determined from Amonton's law, which states that with the addition of a frictional force \( Q \), a tangential force at the contact results that is proportional to the normal force, \( F \), when two bodies are sliding with respect to each other.

\[
Q = \mu F
\]  

(5.3)

The tangential force is opposite the direction of slip. In zones where the tangential force is less than the limiting value of \( \mu F \), a condition known as stick or partial slip exists.

The size of the stick and slip zones is not known in advance, and must be determined by trial-and-error. The procedure used to determine the size of the stick and slip zone is covered in detail by Johnson (1985) and by Hills and Nowell (1994). The evolution of hysteresis with increasing slip amplitude is illustrated in Figure 5.9. When a contact is in the stick condition, no relative slip occurs in a cycle. If the contact is in partial slip for part of the cycle, a small amount of relative slip can be observed in the hysteresis loop, similar to that shown in Figure 5.9 (a) when \( \delta_s = 15 \mu m \). As the amount of slip increases, the hysteresis at the contact also increases. When \( \delta_s = 60 \mu m \) or 120 \( \mu m \), as shown in Figures 5.9(c) and (d), respectively, the contact conditions are those of a gross slip contact condition throughout the test. Note that the slope in the loading/unloading portion of the curve remains about the same throughout the test, which is an indication of the stiffness/compliance of the contact and the fretting apparatus. In other words, the true relative slip at the fretting pad is different than the displacement measured by the clip gage due to the stiffness/compliance of the fretting apparatus.

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The partial slip/stick contact condition is most often associated with the formation of cracks due to fretting and so is usually considered the most damaging contact condition. The gross sliding contact condition most often leads to significant wear, so damage is usually indicated by material removal rather than by the formation and growth of cracks.

The evolution of frictional force versus displacement for Test 3 ($\delta_b = 60 \mu m, N = 10^4$ cycles) is shown in Figure 5.10. Within the first 10 cycles, the contact conditions change dramatically (Figure 5.10 (a)). First, peak-valley compensation was employed in the test control in order to insure that the desired applied maximum and minimum displacement was reached throughout the test. For all tests, the displacement stabilized between the 10th and 30th cycle. Also, recall that all of the pads were machined using an electrical discharge machining (EDM) process and were not polished in order to avoid modifying the pad radius. A summary of measured scar width for each test is given in Table 5.6. The measured scar width after fretting tests is, on average, about four times larger than the theoretical scar width calculated using Hertzian theory (see Table 4.5). However, this is consistent with scar width predictions based on contact between rough surfaces, as described in Chapter 4.

A “bedding” in at the contact occurs, which is similar to that observed in prior fretting fatigue tests conducted on Ti-6Al-4V (Wallace and Neu, 2003). In those tests, the frictional force range reached a steady state condition after about 100 cycles. Refer to Wallace and Neu (2003) and Wallace (2000) for more information about typical fretting response observed in frictional force range plots for Ti-6Al-4V. Fayeulle et al. (1993)
also discuss in detail the evolution of fretting observed by hysteresis loops in tests conducted on Ti-6Al-4V, a near α-titanium, and a pure α-titanium.

For the test condition shown in Figure 5.10, the contact is considered to be in gross sliding throughout the test. In every case, the maximum and minimum load is unsymmetric. The maximum compressive force is consistently higher than the maximum tensile force, on the order of approximately 200 N to 400 N. This phenomenon occurred even though both the force and the clip gage displacement were zeroed prior to beginning the test. Since this was observed in every test, it appears to be an artifact of the fretting apparatus configuration. After a few tests were completed, the grip and actuator were observed to be slightly misaligned. Once the grips were re-aligned, the minimum and maximum tensile force became somewhat more symmetric but was never completely eliminated. It appears that the asymmetry is due to misalignment between the grip and the actuator, along with some misalignment in the apparatus.

There was one test in which the frictional force range was not typical of those observed in fretting experiments. The frictional force range for Test 3 (δ = 60 μm, N = 10^5) shown in Figure 5.11 indicates that it never truly reaches a steady state condition. Instead, the frictional force range appears to be steadily increasing with the number of cycles. Also note the drop in frictional force range that occurred at various points during the test. Interestingly, these drops occurred when there was an increase in δ during the test, as shown in Figure 5.12. The fluctuation in the controlled variable, δ, was attributed to variations in the hydraulic pressure and/or electrical signal supplying the servo-hydraulic test machine that occurred when multiple tests were conducted in MPRL. Specifically, a large 100 kip tension/torsion machine was being operated by another user.
while this test was being conducted. Therefore, subsequent fretting tests were conducted at night and on weekends to avoid “cross talk” between machines.

The evolution of frictional force versus displacement for a Ti-5Al-2.5Sn specimen (Test 14: $\delta_s = 60 \mu m$, $N = 10^4$ cycles) is shown in Figure 5.13. The hysteresis loops for the first 10 cycles in this Ti-5Al-2.5Sn test appear much more stable than the first 10 cycles observed in the Ti-6Al-4V test previously shown in Figure 5.10. The Ti-5Al-2.5Sn specimen was fretted with a Ti-6Al-4V pad, which is slightly harder than the Ti-5Al-2.5Sn. It is possible that there is slightly less adhesion between the two surfaces early in the cycling. Also, frictional behavior is influenced by material texture (Farhat, 2001). It is likely that texture plays a role in rationalizing differences between hysteresis loops observed in the early portion of the fretting tests for the two materials. It should be noted that the grip had been re-aligned prior to beginning the tests on Ti-5Al-2.5Sn. Observe that the asymmetry in the frictional force is somewhat improved in the early portion of the test.

The evolution of frictional force versus displacement for a CP-Ti specimen (Test 22: $\delta_s = 60 \mu m$, $N = 16^6$ cycles) is shown in Figure 5.14. Interestingly, the hysteresis loops for CP-Ti indicate that the response stabilized rather quickly compared to Ti-6Al-4V and Ti-5Al-2.5Sn. In particular, there appears to be less sliding than Ti-5Al-2.5Sn in the early portion of the test (i.e. less than 100 cycles). Note that by the 10,000th cycle, the hysteresis loops appear very similar for each material, which may be an additional indication that an oxide layer has formed for these test conditions. Hysteresis plots for other fretting tests are shown in Appendix B. At every slip amplitude, the hysteresis loops for Ti-5Al-2.5Sn and CP-Ti for cycles 1-10 are consistently less “scattered” than
those observed for Ti-6Al-4V, the implications of which will be discussed in Section 5.3.5.

No major differences were observed in the scar surfaces for each material at a given slip amplitude. Changes in scar morphology, regardless of material, were much more dependent upon the applied slip amplitude. The scar surface for fretting tests with \( \delta_s \leq 30 \mu m \) were dominated by ploughing of asperities. Wear was observed only in isolated locations. In fretting tests with \( \delta_s \geq 60 \mu m \), the scar surface showed evidence of significant wear, ploughing, and pile up, which is consistent with observations of Ti-6Al-4V scar surfaces by Fayeulle et al. (1993) and Sauger et al. (2000).

The overall coefficient of friction, \( \mu \), calculated using frictional force data at cycle 10 and \( \delta_s = 60 \mu m \) is given for all three materials in Table 5.7. The highest coefficient of friction is observed in CP-Ti, most likely due to the considerable ductility this of the specimen compared to the Ti-6Al-4V fretting pad. Ti-5Al-2.5Sn has the lowest coefficient of friction. The hysteresis loops showed that considerably more sliding occurred within the first 10 cycles in this specimen compared to Ti-6Al-4V and CP-Ti. The coefficient of friction in Ti-6Al-4V at the 10th cycle is consistent with previous fretting fatigue tests (Wallace and Neu, 2003).

5.3 Characterization of Fretting Damage

Subsurface damage due to fretting can, in part, be characterized by observation of changes in grain morphology and composition near the fretting contact. Rotation of “markers”, flow, and flattening of grains are all indicators of significant plastic deformation (Rigney, 2000). These changes are most clearly observed in SEM
micrographs of the fretting scar cross sections and in orientation and pattern quality maps obtained using EBSD.

Other indications of fretting damage include oxidation of near surface material, changes in mechanical properties such as hardness, and the development of a preferred orientation or texture of near surface grains. Results of energy dispersive X-ray (EDX) analysis as well as nanoindentation tests will also be shown in order to determine if changes in composition or hardness occur in material very near the fretting contact. Pole figures obtained from EBSD analysis of near surface material will be shown to illustrate changes in texture within the region most likely to be affected by fretting loading.

Recent developments in TEM and SEM technology have allowed researchers to investigate intergranular material response to deformation and elucidate patterns in subgrain formation. Relationships have been derived between misorientation distribution and total or plastic strain (Hughes et al. 1996; Hansen et al. 2001; Angeliu et al., 2000; Butler et al., 2002). For that reason, considerable attention is devoted to the misorientation distribution results obtained by EBSD. The emphasis in this case is determining overall patterns in misorientation distribution in order to shed more light on the potential for using EBSD to improve procedures used to evaluate microstructure evolution and also to validate computational crystal plasticity models used for fretting life prediction.

5.3.1 Ti-6Al-4V

To aid comparisons between the SEM micrographs and texture analysis results discussed in this section, a schematic illustrating the scar orientation relative to the Ti-6Al-4V specimen/plate normal direction (ND), rolling direction (RD), and transverse
direction (TD) is shown in Figure 5.15. All EBSD results for Ti-6Al-4V are oriented according to this convention.

The images of the fretting scars on the Ti-6Al-4V fretting specimens were scanned for reference purposes (Figure 5.16).

An SEM micrograph of a Ti-6Al-4V specimen (Test 2: \(\delta_0 = 60 \mu m, N = 10^5\) cycles) near the edge of the fretting contact and at the center of contact is shown in Figure 5.17 (a) and (b), respectively. Small cracks around 10 \(\mu m\) in length are observed to form near the edge of the contact at a relatively low number of cycles (\(N = 10^5\) cycles). The \(\beta\) phase is a convenient marker, enabling one to observe the grain flattening and "flow" in the grains near the fretted surface. When markers such as these are readily observable, good estimates of displacements, strains, and strain rates can be made (Rigney, 2000; Rainforth, 2000). This type of behavior is not seen immediately outside the contact, on the left of the ridge in Figure 5.17 (a). Here the grains are still roughly equiaxed. There appears to be a distinct change in microstructure near the center of fretting contact (Figure 5.17 (b)). From this image, three distinct layers can be identified. These layers are perhaps more clearly observed in Figure 5.18. The characteristics that distinguish these layers are described as follows:

- Nearest the surface, a layer forms that appears porous and brittle and is often filled with microcracks and voids. It is considered to be an oxidized layer because the concentration of oxygen is highest in this area.
- Directly beneath the oxidized layer, an intermediate or transitional layer may exist that is characterized by severe plastic deformation as indicated by pile up of what appear to be flattened grains of indeterminate size. Some investigation
concerning the morphology of a transitional layer of this nature observed to form in titanium has been performed by others using TEM (Sauge et al., 2000). In the current study, the oxygen concentration here is observed to be much less than in the oxidized region above it, but still relatively high, hence the term "transitional" layer. A transitional layer is not always visually observed.

- The microstructure in the next layer appears similar to the virgin microstructure, though grains have been distorted, likely due to large plastic deformation. This region, termed a fretting disturbed layer is often visually distinguished by flow or rotation of grains and/or formation of primary cracks growing into the specimen. The concentration of oxygen found in this region often, but not always, drops to a level that is not much higher than the concentration found in the virgin material.

- The bulk material region is the area in which no visible evidence of plastic deformation is observed, nor is there any evidence of an increase in oxygen concentration in this region. The bulk material is assumed to have the same mechanical properties and composition as the virgin material.

For the sake of convenience, the oxidized layer will be labeled "3", the intermediate layer labeled "2", and the fretting disturbed layer labeled "1" in cases where the delineation between these layers is quite obvious. Energy dispersive X-ray analysis was conducted in order to more easily distinguish the regions in which substantial oxidation had occurred near the fretted surface (Figure 5.18). The concentration is represented as "counts" along a line. The counts of each element are then integrated in the spectrum analysis to determine the total atomic or weight percent of each element. A higher number of counts for oxygen indicated a higher concentration in that area.
The only elements analyzed in these tests were oxygen, titanium, aluminum, and vanadium. Trace amounts of other elements, such as fluorine, carbon, and silicon were found. The existence of silicon and fluorine is attributed to the fact that the specimen had been polished with colloidal silica and etched with hydrofluoric acid. These elements were therefore eliminated from the spectrum analysis. Details of the spectrum analysis that correspond to each EDX test are located in Appendix D.

As expected, the material closest to the surface shown in Figure 5.18 is considerably oxidized compared to the material beneath it. This finding is typical when wear mechanisms dominate. An overview of recent progress in microstructural characterization of wear in ductile materials is provided by Rigney (2000). Since the existence and composition of the oxidized layer has been so thoroughly documented in the past, the emphasis here will be primarily on the lower most region (labeled "1" in Figure 5.18), called the fretting disturbed layer, and to a lesser degree, on what is called a transitional layer (labeled "2"). In this case, the thickness of the oxidized layer and intermediate layer are about 25 μm and 15 μm, respectively. However, there are some instances where it is not clear what constitutes a transitional or a fretting disturbed layer, the implications of which will be discussed in detail.

An SEM micrograph of an area near the edge of the contact in a fretting test conducted at the same slip amplitude but higher number of cycles (Test 1: δs = 60 μm, N=10^7 cycles) is shown in Figure 5.19. Here, several cracks of length 20-50 μm in length can be seen. The existence of an oxidized layer that is approximately 20 μm thick is confirmed in EDX results shown in Figure 5.20. Interestingly, for this larger cycle experiment, the oxygen content did not drop off immediately at the boundary between the
oxidized layer and the fretting disturbed layer. No transitional layer is observed. Recall that it was previously shown (Figure 5.18) that the oxygen concentration immediately dropped off once inside a region with distinguishable grains. Therefore, multiple tests were conducted in this area to verify that oxygen had diffused into what appears to be the fretting disturbed layer (Figures 5.21 and 5.22). The oxygen concentration remains considerably high to a depth of at least 15 μm into the fretting disturbed region, even in areas where significant cracking is not observed. Interestingly, the depth of increased oxygen concentration corresponds with the depth of primary cracking observed in this specimen. It may also be possible that the interface between α+β phases in Ti-6Al-4V acts as a path for oxygen diffusion. Glaeser and Lawless (2001) observed a higher concentration of oxygen in the vicinity of primary cracks in fretting specimens. Therefore, it is likely that oxygen diffusion is a contributing factor in the formation of cracks due to fretting.

A very thin oxidized layer less than 10 μm thick was observed in a test with lower slip amplitude (Test 5: δs = 30 μm, N=10^5 cycles). There was no evidence of an oxidized or transitional layer at the lowest slip amplitude (Test 6: δs = 15 μm, N=10^4 cycles). The existence of an oxidized or transitional layer seems to correspond with δs ≥ 60 μm or, in other words, when the fretting contact condition was dominated by gross sliding throughout the test. Also, the thickness of the oxidized layer tended to stabilize between N = 10^3 and 10^4 cycles. For tests conducted under gross sliding conditions and a higher number of cycles, the oxidized layer appears to have peeled off. These results are confirmed by fretting tests conducted by Sauger et al. (2000) in which the thickness of
the oxidized layer observed in titanium specimens tended to increase with slip amplitude, but stabilized between $10^5$ and $10^6$ cycles.

The cracks observed in Figures 5.17 and 5.19 appear to be primarily transgranular and growing through the $\alpha$ phase. Transgranular cracking in $\alpha$ grains has been noted to occur due to cleavage along favorably oriented basal planes (Bache et al., 1995). Cracks of length greater than 10 $\mu$m were not observed in Ti-6Al-4V fretting tests with $\delta_s < 60$ $\mu$m. One very small crack (less than 5-10 $\mu$m) was observed near the edge of contact in a specimen tested at a lower slip amplitude (Test 5: $\delta_s = 30$ $\mu$m, N=$10^4$ cycles). The hysteresis loops for tests conducted at this slip amplitude indicate contact conditions in which partial slip/stick are dominant. This observation further tends to support the hypothesis that oxygen diffusion plays an integral role in the formation of cracks in the fretting affected region since the only area where significant cracks of length greater than 10 $\mu$m were observed also contained a substantial concentration of oxygen. It is likely that the magnitude of the slip amplitude plays a role in enhancing oxygen diffusion into the fretting disturbed layer.

Nanoindentation tests were conducted to shed light on possible changes in mechanical properties that may occur in the very near surface material due to fretting loading. Since titanium is highly anisotropic at the scale of the grains, the depth of indents differs dramatically when nanoindentation tests are conducted under load control. Therefore, all indentation tests were conducted in displacement control using the continuous stiffness mode (CSM). Before conducting an actual test, it is necessary to determine the depth at which the hardness and elastic modulus tend to stabilize. If the indent is too shallow, then near surface effects related to polishing and so forth will cloud
the results. In Ti-6Al-4V, a depth greater than 200 nm was sufficient to obtain stable hardness and elastic modulus measurements, so all tests were conducted at a depth of 500 nm. Also recall that the elastic modulus obtained in an instrumented nanoindentation test is traditionally based on elastic, isotropic material assumptions. If the elastic modulus from a nanoindentation test is desired for titanium, it would need to be corrected using the anisotropic stiffness matrix.

In tests conducted on Ti-6Al-4V, an array of 10 x 10 indents (100 total) spaced 30 μm x 30 μm apart were placed in the sectioned sample near the fretted contact. Each row of indents was averaged and compared to the average hardness from rows of indents placed similarly in a virgin Ti-6Al-4V specimen. A summary of results obtained on a fretting specimen tested at δₜ = 60 μm and 10⁹ cycles (Test 1) are listed in Table 5.8. The first row of indents were eliminated from the data set because they fell into the specimen mount. The average hardness for each row in the fretted specimen is softer than the average hardness in the virgin Ti-6Al-4V to a depth of approximately 150 μm. Some additional observations can be made by examining selected indents from this series of tests highlighted in Figure 5.23(a), which show part of rows 2, 3, and 4. Compare the values shown here with the average hardness for all indents in the virgin specimen, which is 4.32 GPa. While indents 1 and 3 indicate areas that are slightly harder than the average hardness in the virgin material, it appears that the indents are near grain boundaries and/or near α+β lamellae.

Nanoindentation tests were also conducted near the fretted surface in specimens tested at the same slip amplitude, but a fewer number of cycles (Test 2: δₜ = 60 μm and 10⁹ cycles; Test 3: δₜ = 60 μm and 10⁹ cycles). In both cases, the material near the fretted
surface was 3% and 22% softer, respectively, than the virgin material. The literature tended to indicate that the material very near the fretting contact should be harder, not softer, than the virgin material (Sauger, et al., 2000). For this reason, additional indentation tests were done to isolate regions of varying hardness within the transitional layer, as shown in Figure 5.23 (b) for Test 2 (δ₀ = 60 μm, N=1000).

Indents numbered 6, 9, 10, and 11 have a hardness exceeding 5 GPa. Indents 9 and 10 located within the transitional layer show the greatest hardness values; 6.53 and 6.20 GPa, respectively. Note that indent 13, which is also located within this area, is only 4.37 GPa—almost equal to the average hardness found in the virgin material. The oxidized and transitional layers apparently contain pockets of material that are harder than the bulk material as well as areas with much lower hardness due to multiple cracks and perhaps porosity. This would explain why microhardness tests using a much larger Vickers indenter would indicate a higher hardness in this region. However, average results for rows of indents placed in the fretting disturbed layer immediately below the oxidized/transitional layers consistently indicates material softening. This is significant because primary fretting cracks are observed to nucleate and grow in this region.

This region experiences cyclic plastic strain accumulation and Ti-6Al-4V cyclically softens. This may explain why the material within the fretting disturbed region in Ti-6Al-4V would also soften. It is possible that compliance near the edge would affect the hardness measurements, but this would not explain why the material is consistently softer to a thickness extending about 3 to 5 grains deep.

According to Hirth and Rigney (1976), the tendency of ductile materials to harden or soften near a surface subjected to sliding depends on the stacking fault energy of the
material. The scale at which these changes occur is estimated to be on the order of 1-3
grain diameters early in deformation, or around 1-100 μm in depth. Interestingly, this is
about the same scale of softening observed in Ti-6Al-4V at the surface. However, only
materials with lower stacking fault energy are expected to experience near surface
softening compared to materials with high stacking fault energy when subjected to sliding
loads. Titanium is considered to be a fairly high stacking fault energy material (Tan and
Gu, 1995).

Another possible explanation may be that fretting induces a preferential grain
orientation, or texture, near the surface such that the indent direction is parallel to the
"softer" direction in the HCP unit cell. For instance, if a basal texture formed (basal
normal parallel to Z(ND)) due to fretting, then indents placed perpendicular to the HCP
unit cell (perpendicular to [0001] direction) would promote slip on the prism planes. The
prism planes have the lowest critical resolved shear stress compared to other planes,
making it more favored for slip (Feagus et al., 1997a). This topic will be reintroduced
later after texture analysis results are presented.

For the EBSD results that follow, an orientation map legend is provided that link
colors with Euler angle directions (Figure 5.24). EBSD results of a specimen tested at an
intermediate slip amplitude and higher number of cycles (Test 1, δ = 60 μm, 10^5 cycles)
are shown in Figure 5.25. A map representing α phase grain orientations via Euler angles
(Bunge convention) is shown in Figure 5.25 (a). A pattern quality map, which appears
similar to an SEM micrograph, is shown in Figure 5.25 (b). The standard convention for
defining a grain boundary is a misorientation of at least 10° or 15° (HKL Technology,
2000). From these two images, one can clearly see that the grains within the fretting

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disturbed layer (about 20 to 40 \( \mu m \) deep) are significantly deformed and are much smaller and flatter than grains farther away from the surface. An inverse pole plot (Figure 5.25 (c)) is provided to help locate poles relative to specific directions in the HCP unit cell.

The tendency for grain refinement in the fretting disturbed layer can be seen at a relatively low number of cycles \((N = 1000)\), as shown in Figure 5.26 (b) and (c). An SEM micrograph of the area examined with EBSD is shown in Figure 5.26 (a) to relate the pattern quality map to the oxidized and/or transitional layer. Scattered indexation indicates a random orientation with a scale that is much smaller than the step size (typically 1.5 to 2 \( \mu m \)) for any given EBSD experiment. The noise reduction algorithm in the postprocessing software (HKL, Technology, 2000) filters out orientation measurements in this region as “wild spikes”. TEM analysis conducted on very near surface layers in titanium indicate a very fine grained microstructure with grain sizes on the order of a few tens of nanometers (Stauger et al., 2000). The resolution required to accurately index grains of this size is beyond the capability of the current EBSD system at Georgia Institute of Technology. It should also be noted that no Kikuchi patterns were visually observed in this region, which is another indication of significant plastic deformation. Recall that Kikuchi patterns are very sensitive to lattice distortion resulting from plastic deformation (Randle and Engler, 2000).

EBSD not only aids studies of grain morphology within the fretting disturbed region, but it is also a very useful tool to determine whether texture forms due to fretting along with providing clues concerning the deformation substructure. The depth of material sampled for texture and misorientation distribution analysis was determined
based on observations of the SEM micrographs, EBSD orientation maps and EBSD pattern quality maps shown previously. Whenever possible, at least 100 grains were sampled for each pole plot, at a depth that is equal or greater than the maximum grain diameter in the as-received material. However, note that even for a relatively small sampling of grains (i.e., less than 50), the number of orientation measurements was always more than 800. As few as 100 orientation measurements are required to detect a moderate degree of texture (Wright and Adams, 1990).

In Ti-6Al-4V, the average grain diameter is approximately 10-15 μm with a maximum grain size of approximately 40 μm, so the texture analysis was conducted at a depth between 30 to 50 μm within the fretting disturbed region. Pole figures are plotted according to standard convention, with the basal plane normal [0001] ideal orientation rotated parallel to the Z (ND) direction in the plate/specimen (see Figure 5.15). When appropriate, the texture will be classified as basal, transverse, or basal/transverse. However, texture results do not always fall into these categories. For that reason, the angle, θ, of the basal plane normal [0001] relative to the Z (ND) direction is defined for every case in which the minimum uniform density (MUD) exceeds an arbitrary value of about 5. If the poles fall along the RD or TD axis, this is also noted.

The pole figure shown in Figure 5.27 illustrates the texture in a longitudinal cross section of the as-received Ti-6Al-4V plate, where $D_{\text{max}}$ is the maximum grain diameter and $N_g$ is the number of sampled grains reported by Channel 5 (HKL Technology, 2000). This pole figure of this cross-section represents texture in the same orientation as the fretted specimens. Therefore, it can be used to determine if texture develops due to fretting.
In the as-received material, the poles have low intensity of about 3 to 4, and are randomly dispersed, which indicates a relatively weak texture. The random or weak texture in the as-received specimen is confirmed by the misorientation distribution plot shown in Figure 5.28. Weak or random texture is associated with close correlation with the theoretical distribution. The theoretical random distribution for an HCP material (Grimmer, 1979) is shown as a continuous solid line in these plots. The Y-axis is the relative frequency of misorientations occurring at angles binned at 5° increments, starting at around 2.5°, which is plotted on the X-axis. In particular, note that the relative frequency of misorientation angles less than 5° is similar to the theoretical random.

The box on lower portion of the misorientation plot indicates the number of points sampled and whether the data is correlated (near each other in the sample area) or uncorrelated (randomly located). At least 2000 uncorrelated misorientation angle measurements were sampled at random within the subset of grains previously selected for the texture analysis within a depth of 30 to 50 μm in the fretting disturbed region.

Figure 5.29 shows the influence of number of cycles on texture for tests conducted at δ = 60 μm. A much stronger pole density, or intensity, compared to the as-received Ti-6Al-4V is observed. When N ≤ 10⁴ cycles, the poles are rotated between 70° to 80° from the Z(ND) direction. In two cases (Figures 5.29 (a) and (c)), the poles have rotated towards the X(RD) direction. By 10⁵ cycles, the pole figure indicates that a relatively strong basal texture has developed due to fretting. A summary of texture analysis results, including the maximum grain diameter (Dmax), number of sampled grains (N0), the number of orientation measurements (Nα), and observed texture in each pole.
plot is given in Table 5.9. The maximum grain size tends to decrease with increasing number of cycles.

A texture component map (Figure 5.30) clearly shows the orientation of grains within the fretting disturbed region for the test in which a strong basal texture was indicated in the pole figures. (Test 1; \( \delta_0 = 60 \) \( \mu \)m, \( N=10^5 \) cycles). The inverse pole plot indicates that grains colored red have basal plane normals oriented nearly perpendicular to the fretted surface, meaning that the basal planes are nearly aligned with the surface. A grouping of grains with basal plane normals oriented less than 30° from the Z(ND) direction near the surface confirms that a basal texture has formed in near surface grains due to fretting in this specimen.

Misorientation distribution histogram plots in Figure 5.31 show the effect of an increase in cycles when \( \delta_0 = 60 \) \( \mu \)m. The sample area of the histogram plots is identical to the sample area for the pole plots used for texture analysis. The relative frequency of misorientation angles between 0° to 10° has at least doubled compared to the virgin material (Figure 5.28). Spikes in the relative frequency of misorientation angles of 20° to 30° and 60° to 70° degrees are also routinely observed.

Pole figures showing the influence of relative slip amplitude for fretting at \( 10^4 \) cycles is shown in Figure 5.32. A very strong basal texture with the [0001] poles oriented at 0° to 15° relative to the Z (ND) direction clearly develops at the highest strain amplitude (\( \delta_0 = 120 \) \( \mu \)m). Note that the strongest pole intensity is located along the X(RD) axis. At lower strain amplitudes (\( \delta_0 = 15, 30 \) \( \mu \)m), there appears to be texture with [0001] poles oriented at 55° to 65° relative to the Z (ND) direction. A grouping of poles at angles higher than 80° relative to the Z(ND) direction, such as those shown in Figure
5.32 (c) are considered to be more indicative of transverse texture. A texture component map of the $\delta_0 = 120 \mu m$ experiment is shown in Figure 5.33. A group of grains within about 30-40 $\mu m$ in the fretting disturbed layer have a basal plane normal angle less than about 15° relative to the Z(ND) direction.

A summary of texture analysis results for the pole plots shown in Figure 5.32 is given in Table 5.10. The misorientation distribution histogram plots that correspond with the sample area used for these pole plots are shown in Figure 5.34. The trend in the distribution is similar to the results shown in Figure 5.31. A significant increase in low angle misorientations is observed as well as spikes in the angle near 60° to 70°, especially in the $\delta_0 = 120 \mu m$ experiment.

Significant primary cracks of length greater than 10 $\mu m$ were observed in only two fretting tests in Ti-6Al-4V (see Figures 5.17 and 5.19). Interestingly, both tests were conducted at $\delta_0 = 60 \mu m$. The largest fretting cracks (20 to 50 $\mu m$) were observed for the test with the highest number of cycles ($N = 10^5$), which corresponded with a strong basal texture (Figure 5.29 (c)). However, large cracks were not observed in specimens tested at $\delta = 120 \mu m$ and $N = 10^4$ cycles, even though a strong basal texture was observed. A possible explanation may be that above certain slip amplitudes, any cracks that form may be quickly obliterated by the reciprocal sliding motion. Also, more cycles may be required for crack formation. Below certain slip amplitudes and number of cycles, a strong basal texture may not form. In other words, there appears to be a critical threshold in slip amplitude and number of cycles in which mechanisms such as oxidation and grain reorientation combine to create conditions that are ideal for crack formation. For instance, consider the results of fretting only tests of Ti-6Al-4V conducted by Glaeser.
and Lawless (2001). Cracks due to fretting only were consistently observed in tests conducted with $\delta_n = 38 \mu m$ at a particular normal load. The trend in crack formation was much less clear in specimens fretted at either a lower slip amplitude of 20 $\mu m$ or higher slip amplitude of 75 $\mu m$. Cracks were only observed in isolated tests at those slip amplitudes. It should also be noted that all of these fretting tests were conducted until $9 \times 10^3$ cycles. Grain diameter can most likely be ruled out as a factor when comparing the current results with those of Glaeser and Lawless (2001), as both groups of tests were conducted using the same pedigreed Ti-6Al-4V used in the Air Force HCF program. They also used flat pads instead of cylindrical pads, which may also be a factor.

There is some debate whether cracks in titanium are more likely to form due to cleavage between favorably oriented basal planes (Bache et al., 1998). Therefore, an EBSD experiment with a smaller step size (0.5 $\mu m$) was conducted for the grains surrounding the crack in order to link grain orientation with the crack plane. Comparison between the SEM micrograph and texture component map shown in Figure 5.35 (a) and (b), respectively, indicate that a group of grains surrounding the crack have basal plane normals oriented within 30° relative to the Z(ND) direction. It is very clear from Figure 5.35 (a) that the crack growth is transgranular, which is supported by Wanhil et al. (1989). A direct link between grain orientation and crack formation is less obvious, however. The crack grows perpendicular to the fretting direction until the grains in the vicinity of grains labeled A and B. Grain A has a basal plane orientation around 30° relative to the Z(ND) direction, while the orientation of Grain B has a basal plane normal oriented around 10° relative to the Z(ND) direction. In this area, the crack appears to grow due to cleavage between the basal planes of Grain A and B. It is not clear how
grain orientation affects the crack growth direction either closer to the surface or after it grows past Grains A and B.

5.3.2 Ti-5Al-2.5Sn

A schematic illustrating the specimen and fretting scar orientation relative to the as-received Ti-5Al-2.5Sn bar normal direction (ND), rolling direction (RD), and transverse direction (TD) is shown in Figure 5.36. Both sides of the Ti-5Al-2.5Sn specimen were scanned for reference purposes (Figure 5.37).

SEM micrographs of fretting damage in a Ti-5Al-2.5Sn specimen (Test 12, δ = ±120 μm, N=10⁵) are shown in Figure 5.38. Some interesting microstructural features observed within this region are highlighted. For instance, flow in the grains near the surface can be easily seen in Figure 5.38 (b) and (c). The maximum combined thickness of the oxidized and transitional layer is approximately 25 μm deep in this test. Three distinct layers with different morphology can be detected, which is similar to the results observed in Ti-6Al-4V. The interface between what is assumed to be the oxidized, transitional, and fretting disturbed layers can be more easily observed in Figure 5.38 (d). The uppermost oxidized region, labeled “3”, appears to be very porous and filled with multiple microcracks. The second, or transitional, layer is characterized by significant layering of grains, which then transitions to a fretting disturbed region, labeled “1”, in which discernable grains are observed. Along the length of the fretting contact, pockets of material with this morphology exist.

An SEM micrograph of a Ti-5Al-2.5Sn test conducted at a lower slip amplitude (Test 14: δₕ = 60 μm, N=10⁶ cycles) is shown in Figure 5.39. One can see that the thickness of the oxidized and transitional layers vary greatly from one grain to the next.
along the surface. Perhaps there are regions (i.e., grains) that are softer or oriented in such a way allowing greater ploughing of the material compared to the surrounding grains. The same pattern, albeit on a smaller scale, was observed in Ti-6Al-4V when $\delta_s \geq 60 \mu m$ and $N \geq 10^7$ cycles (see Figure 5.17 (b)). Interestingly, formation of cracks growing into the fretting disturbed layer were not observed in any of the fretting cross sections in Ti-5Al-2.5Sn even though the same range of slip amplitudes were used in the experiments. Both the Ti-5Al-2.5Sn and Ti-6Al-4V tests were conducted with a normal force ratio corresponding to $F/F_y = 0.05$.

In fretting scar cross sections of Ti-5Al-2.5Sn specimens, increased oxygen concentration was observed in the oxidized and transitional layers (Figures 5.40 and 5.41). The highest oxygen concentration occurs in region “3”, which is closest to the fretting contact, so this is considered to be the oxidized layer. The concentration drops immediately once within the transitional region, labeled “2”, to an intermediate level, then drops further once within fretting disturbed region labeled “1”. Unlike the EDX results shown for fretting tests conducted on Ti-6Al-4V (see Figures 5.20, 5.21, and 5.22) there does not appear to be a significant increase in concentration of oxygen within the fretting disturbed region.

An SEM micrograph, orientation map, pattern quality map, and inverse pole plot obtained near the center of contact in a Ti-5Al-2.5Sn fretting wear (Test 18: $\delta_s = 60 \mu m$, $10^7$ cycles) are shown in Figure 5.42. Comparison between these images illustrates again that the pattern quality map can pick up features such as the oxidized or transitional layers, even when grains in this region are not indexable. Others have used the pattern

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quality maps to determine the size of the plastic zone in a fine grained (i.e., 5 μm) nickel based superalloy (Tucker et al., 2000)

Also note the areas near the surface where grain refinement is most obvious. Specifically, there appear to be pockets of material where the combined oxidation/transitional layer is deeper and where grains are more refined in comparison with the surrounding grains, which was also observed in other tests.

The orientation map, inverse pole plot and pattern quality map for a lower slip amplitude test (Test 16: δs = 15 μm, N =10⁶ cycles) are shown in Figure 5.43. Large grains are filled with regions of material that routinely indicate misorientations near 90° with Miller indices of \([\bar{1}0\bar{1}], [1\bar{1}0], [411],\) or \([4\bar{1}1]_7\). The software allows the user to rotate systematic misindexing to the orientation of the dominate grain, however, a pattern appeared to emerge in these misindexations that should not be eliminated or necessarily assumed to be incorrect. Conversion to Miller-Bravais indices yields \([\bar{1}100], [1\bar{1}00], [72\bar{5}1],\) and \([32\bar{1}1]\). The \([\bar{1}100]\) and \([1\bar{1}00]\) indices correspond to the \([11\bar{2}2]\) family of compressive twins (Yoo, 1981; Salem et al., 2002), however, the misorientation angle of 90° is unexpected. Salem et al. (2003) observed misorientation angles around 63° in CP-Ti specimens tested in compression. The last two sets of indices have not been related to twins. It is possible that these misorientations are actually misindexed beta grains. However, this pattern of misorientations was not observed in the EBSD tests on the as-received material nor in Ti-6Al-4V, which has a much higher percentage of beta phase than Ti-5Al-2.5Sn. If these were simply misindexed beta grains, this pattern would most likely be observed in the virgin material as well. Another explanation may be that
the groups of misorientations are colonies within prior β-grains (PBGs). If the colonies obey the Burgers relation, then PBG sharing colonies will have basal planes at angles of 0°, 60°, and 90° in the ratio of 1:4:1 Wilson et al. (1997).

This pattern of misindexations appeared in many of the EBSD results on Ti-5Al-2.5Sn and CP-Ti. Originally, it was thought to result from very local surface roughness. However, if that were the case, the misorientations would be random and not systematic. Also, the CP-Ti specimen was electropolished, not mechanically polished like the Ti-6Al-4V and Ti-5Al-2.5Sn.

Results of nanoindentation tests conducted on Ti-5Al-2.5Sn are shown in Figure 5.44 and summarized in Table 5.11. The first row of 5 indents were placed 20 microns apart in the oxidized layer. The hardness in this region is considerably greater than the hardness in the fretting disturbed layer directly underneath. While there are isolated locations that have higher hardness, the average of the indents along each row indicates that the material in the fretting disturbed region is softer than the virgin material to the maximum depth tested (about 45 μm). Each row is composed of 20 indents, placed 20 x 20 microns apart. These results are consistent with those obtained in Ti-6Al-4V, in which material within the fretting disturbed region was found to be softer than the virgin material. Ti-5Al-2.5Sn also cyclically softens (Figure 5.4), therefore, the nanoindentation tests confirm the accumulation of cyclic plastic strain.

In Ti-5Al-2.5Sn, the maximum grain diameter in the as-received bar is about 70 μm with an average grain size of approximately 25 μm, therefore, the area sampled for the texture analysis and misorientation distributions ranged between 50-70 μm in the
fretting disturbed layer. Similar to Ti-6Al-4V, the oxidized/transitional layer did not index well.

The pole figure for a longitudinal cross section of the as-received Ti-5Al-2.5Sn bar is shown in Figure 5.45. A strong fiber texture with $\theta \approx 45^\circ$ along the transverse (TD) direction is observed in this case. This bar was rolled along the X(RD) direction and stress-relief annealed. The texture shown here may not seem intuitive for a bar that has been rolled along the X(RD) axis. The examples of texture in rolled plate (see Figure 2.21) show that the HCP unit cell has basal planes oriented parallel to the rolling direction, with the basal plane normal oriented parallel to either the Z(ND) direction or the Y(TD) direction depending upon rolling temperature and rolling direction.

For the sake of comparison, an as-received specimen with random texture, similar to that shown in the as-received Ti-6Al-4V specimen, would have been preferred over a Ti-5Al-2.5Sn specimen with texture this strong. However, obtaining this material in a size suitable for machining specimens was not available.

A plot comparing the misorientation distribution histogram from a cross-section in the longitudinal (RD) direction in the as-received Ti-5Al-2.5Sn bar and a theoretical random distribution is shown in Figure 5.46. The non-random texture of the as-received Ti-5Al-2.5Sn bar is confirmed by the significant deviation from the theoretical random distribution. In this case, the histogram distribution is entirely shifted towards the lower misorientation angles. This sort of shift is observed in OFHC copper that has been significantly deformed in compression (Bergenaut, 2002). However, the relative frequency of low angle misorientations ($<5^\circ$) is not high, which indicates nearly full recovery due to mill anneal process.
Pole figures shown in Figure 5.47 illustrate the effect of increasing cycles when $\delta_a = 60 \ \mu m$ for a region within 50-70 $\mu m$ in the fretting disturbed region. The dominant pole intensity shown in Figure 5.47 (a) indicates that the basal plane normal [0001] has rotated from about 45$^\circ$ along the TD axis in the as-received bar to about 20$^\circ$ and is aligned along the RD axis. Other poles of lower intensity are roughly distributed along the TD axis. The pole figure shown in Figure 5.47 (b) indicates strong pole intensity in multiple locations rotated approximately 30$^\circ$ to 40$^\circ$ around the Z(ND) direction.

A summary of texture analysis results is given in Table 5.12. The maximum grain diameter decreases only slightly with increasing number of cycles, but is much smaller than the maximum grain diameter in the as-received bar.

Pole figures shown in Figure 5.48 illustrate the effect of slip amplitude when $N = 10^7$ cycles in Ti-5Al-2.5Sn specimens. Table 5.13 gives the texture analysis results for this case. The maximum grain diameter decreases somewhat sharply when $\delta_a = 30 \ \mu m$, but is relatively constant for $30 \ \mu m \leq \delta_a \leq 120 \ \mu m$. This trend is similar to the one observed in Ti-6Al-4V fretting tests with change in slip amplitude.

For each test shown here, a strong pole intensity is observed between 65$^\circ$ to 75$^\circ$, usually along the TD axis. It appears that the strong fiber texture seen in the as-received bar (see Figure 5.45) has been dispersed to some degree due to fretting, with a distribution along the TD direction. It is not until $N = 10^7$ cycles ($\delta_a = 60 \mu m$) that a preferential orientation approaching what would be considered a basal texture is observed, as shown in Figure 5.47(b).

Corresponding misorientation distribution histogram plots are shown in Figure 5.49 and 5.50. When the slip amplitude is increased or the number of cycles is
increased, the relative frequency of misorientation angles less than 5° are observed to increase by four times or more compared to the as-received specimen. Otherwise, the overall distribution appears similar to the as-received specimen with one exception. When δ₀ = 15 μm and N = 10⁴ cycles (Figure 5.50 (a)), spikes in the relative frequency of misorientation angles between 80° and 90° are observed. This trend was not detected in any other Ti-5Al-2.5Sn fretting tests.

5.3.3 CP-Ti

A schematic illustrating the scar orientation relative to the CP-Ti specimen/plate normal direction (ND), rolling direction (RD), and transverse direction (TD) is shown in Figure 5.51. All SEM micrographs and EBSD results on CP-Ti conform to this convention.

Both sides of the CP-Ti fretting specimen were scanned for reference purposes (Figure 5.52). The CP-Ti specimen shown here had been sectioned prior to scanning.

SEM micrographs of the fretting cross section of CP-Ti Test 23 (δ₀ = 120 μm, N=10⁴ cycles) are shown in Figure 5.53. The combined oxidation and transitional layer is at least 50 μm thick in some areas (Figure 5.54). Very large pockets of transitional material with a thickness equal to at least 30 μm exist within the oxidized layer. This transitional material, in CP-Ti as well as Ti-6Al-4V and Ti-5Al-2.5Sn, are all characterized by evidence of severe plastic deformation and layering/pileup of thin bands. This region was often less than 10 μm thick for Ti-6Al-4V, but a single pocket of thickness equal to about 15-20 μm was observed in Test 2 (δ₀ = 60 μm, N=10⁴ cycles). For CP-Ti and Ti-5Al-Sn there were many pockets of transitional material with a thickness of 10 μm to 30 μm observed within the oxidized layer.
EDX results shown in Figures 5.54 and 5.55 confirm that the oxygen concentration is the highest in the uppermost, oxidized layer and drops immediately once within the fretting disturbed region, which is characterized by discernable grains. This trend is similar to the EDX results observed in Ti-5Al-2.5Sn. A trace amount of aluminium and vanadium was detected within the oxidized/transitional region indicating material transfer from the Ti-6Al-4V pads and subsequent mixing with the CP-Ti oxidized layer.

Recall from Chapter 4 that the CP-Ti specimens had to be electropolished in order to obtain EBSD patterns, which necessitated mounting in a non-conductive material. To avoid excessive charging in the SEM, the specimen was removed from the mount to perform EBSD experiments rather than applying a light carbon coating. Unfortunately, the oxidized and transitional layers were often peeled off when the specimen was removed from the mount. Without the oxidized/transitional layer in place, it can be somewhat difficult to locate the scars. As a result, the EBSD was performed in such a way that the fretting scar could be seen when the specimen was tilted in the SEM. This turned out to be especially useful considering the fact that details on the scar face could also be viewed while setting up the EBSD experiment. This configuration requires that the specimen be viewed “upside down” because of the high tilt angle. Therefore, EBSD results for CP-Ti show the fretted surface located at the bottom of the orientation and pattern quality maps. For example, an orientation map, inverse pole plot, and pattern quality map on CP-Ti (Test 21, \( \delta_s = 60 \, \mu m \), 10^3 cycles) are shown in Figure 5.56 (a), (b), and (c), respectively. The grains are so large (\( D_{max} \approx 200 \, \mu m \)) in CP-Ti that nearly the
entire fretting scar could be captured in these EBSD tests. However, a larger step size around 5 µm was used to limit the time of the run.

The EBSD results on CP-Ti are somewhat similar to the results on Ti-5Al-2.5Sn. There appear to be pockets of refined grains separated by larger grains nearest the surface in the fretting disturbed layer. It also appears that twinning has occurred near the surface. The misorientations of possible twinned areas are consistently about 63° (Figure 5.57(a)), which directly correspond to twinning misorientations observed in compression tests conducted on CP-Ti by Salem et al. (2003). Also, the axis of rotation indicates twinning in the [1122] family of compressive twins, similar to those observed by Salem et al. (2003) in CP-Ti. The line in the orientation maps (Figure 5.57(a)) indicate the misorientation region. Recall that twins represent a mirror image of the lattice in the untwinned material. This is seen more clearly in the inverse pole plot shown in Figure 5.57(b). The poles of the grains and possible twins plot in nearly the same location.

An SEM micrograph indicating the two areas selected for nanoindentation of a CP-Ti fretting specimen is shown in Figure 5.58. Two areas were selected for discussion here. A 20 x 3 matrix of indents, spaced 20 µm apart, were placed near the surface. A few indents in the first row fell within the oxidized/transitional layer, while others fell within the fretting disturbed layer. The second and third rows all fell within the fretting disturbed layer. Ten indents each from these three rows are shown in Figure 5.59. Near the interface between the oxidized layer and the fretting disturbed layer, the indents indicate material that is much harder than the virgin material. When all twenty indents in a row are averaged, it appears that the fretting disturbed layer in CP-Ti is harder than the
virgin material. A summary of these results is given in Table 5.14. Recall that the cyclic stress-strain plot (see Figure 5.4) indicates that CP-Ti tends to cyclically harden.

Selected indents made in the oxidized layer, as shown in Figure 5.60, show that the material in this region is also much harder than the fretting disturbed region or the virgin material. This corresponds with results from Ti-6Al-4V and Ti-5Al-2.5Sn, however, in those materials it was shown that the material in the fretting disturbed layer tended to soften. Therefore, nanoindentations within the fretting disturbed layer in Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti indicate significant cyclic plasticity in this region that correlates with bulk material cyclic behavior.

The texture analysis and misorientation distributions were obtained from a sample area 200 to 220 μm within the fretting disturbed region. A pole figure showing texture in the as-received CP-Ti plate in the same direction as the fretting specimen (longitudinal cross section) is shown in Figure 5.61. A strong fiber texture with θ = 45° along the transverse (TD) direction is observed in this case. A secondary pole is observed at θ = 75°, also along the transverse (TD) direction. The texture in the as-received CP-Ti plate is similar to the texture in the as-received Ti-5Al-2.5Sn bar. Recall that both the CP-Ti plate and the Ti-5Al-2.5Sn bar were rolled along the X(RD) axis, then stress-relief annealed. The misorientation distribution (Figure 5.62) indicates a deviation from the theoretical random distribution, and a peak in low-angle misorientations less than 5° indicating that the annealing process did not result in complete recovery.

Pole figures of the fretting disturbed layer are shown in Figure 5.63. These pole figures show the effect of increasing number of cycles when δₙ = 60 μm. A summary of the texture analysis results for this case is given in Table 5.15. Pole figures showing the
effect of slip amplitude for \( N = 10^4 \) cycles are shown in Figure 5.64 and are summarized in Table 5.16. The CP-Ti maximum grain diameter within 200-220 \( \mu m \) in the fretting disturbed layer tends to decrease by about 13% when either the number of cycles increases or for an increase in slip amplitude.

The texture appears relatively unchanged when fretted at \( \delta_s = 60 \mu m \) for \( 10^1 \) cycles shown in Figure 5.63 (a). However, there is some indication that the poles are more distributed along the TD direction than in the as-received specimen. The dominant pole intensity shown in Figure 5.63 (b) after \( N = 10^4 \) cycles indicates a rotation towards \( \theta \approx 70^\circ \), which is nearly a transverse texture as defined by Peters et al. (1984). Note that there are relatively few poles near the center of the plot, which is a further indication of a mostly transverse texture.

For each CP-Ti test shown here, a strong pole intensity is observed between 65\(^\circ\) to 70\(^\circ\), usually along the TD axis. It appears that the strong fiber texture seen in the as-received plate (see Figure 5.61) has been dispersed to some degree due to fretting, with a distribution along the TD direction. The changes in texture that occur due to a CP-Ti fretting test variation in either the number of cycles or slip amplitude resemble the Ti-5Al-2.5Sn texture results shown previously. Neither material exhibits significant changes in texture with either an increase in number of cycles or an increase in the slip amplitude. It should be pointed out that the fretting contact half-width is about 97 \( \mu m \) in CP-Ti and about 138 \( \mu m \) for Ti-5Al-2.5Sn. The maximum grain size in CP-Ti is of the same order of magnitude as the fretting contact half-width, meaning that most of the damage could theoretically be isolated to as few as one grain deep.
Corresponding misorientation distribution histograms shown in Figure 5.65 and 5.66 indicate that the relative frequency of misorientation angles between 0° to 5° in the fretting disturbed layer increase by four times or more compared to the as-received specimen. Otherwise, the overall distribution tends to be scattered with no clear pattern or preferred misorientation angles. This may be due to the fact that the CP-Ti maximum grain diameter in the as-received plate (D_{max} = 200 \mu m) is larger than the fretting contact half-width (a = 97 \mu m).

5.3.4 Texture and Misorientation Comparison

The overall trend in misorientation distribution for all three materials is more easily distinguished when the average relative frequency in misorientation angles for a number of fretting tests are plotted together, as shown in Figure 5.67. The reason for plotting all of the fretting tests together for a particular material is to determine whether spikes in certain misorientation angles are observed regardless of fretting test condition such as the number of cycles or the slip amplitude. If so, this may lend credence to the hypothesis that the average misorientation angle can be used as a scaling parameter to determine the amount of plastic strain. However, the best method to determine the average misorientation is still debatable.

Comparison with the theoretical random distribution shown in this plot indicates that, irrespective of slip amplitude and number of cycles, the misorientation distribution routinely spikes at angles ≤ 5° for all materials, between 20° to 30° for CP-Ti and Ti-5Al-2.5Sn, and near 67° to 70° for Ti-6Al-4V. The relative frequency of misorientation angles between 40° and 50°, and between 70° to 90° are lower than the theoretical random distribution.

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The average misorientation distribution of Ti-5Al-2.5Sn and CP-Ti appear similar even though the distribution observed in individual tests appear much more scattered. While the initial texture in CP-Ti and Ti-5Al-2.5Sn were similar, the misorientation distribution of the as-received CP-Ti plate indicated that it had less texture than the Ti-5Al-2.5Sn bar. The spike in average misorientation angle at 30° to 35° is nearly identical in CP-Ti and Ti-5Al-2.5Sn. This spike was not observed in the as-received CP-Ti or Ti-5Al-2.5Sn. The distribution for angles less than 30° appear similar for all three materials, the Ti-6Al-4V distribution deviates from the other two materials most noticeably at angles between 60° to about 80°.

In Ti-6Al-4V, an increase in low angle misorientation (<5°) and an increase in the relative frequency of misorientation angles around 65° appears to be a direct result of fretting. The increase in relative frequency of misorientation angles <5° and around 30° are linked to fretting in CP-Ti and Ti-5Al-2.5Sn. The fact that all materials exhibit a significant increase in low angle misorientations seems to indicate that subgrain formation occurs due to fretting. The increase in low angle misorientations was observed for all fretting tests shown here and every material, regardless of applied slip amplitude and number of cycles.

The maximum grain diameter was chosen for comparison purposes instead of the average grain diameter because the average grain diameter measured using Channel 5 software (HKL Technology) seemed to be more dependent upon the indexation rate and noise reduction algorithm. The effect of an increase in cycles on maximum grain diameter when δ_n = 60 µm for all three materials is plotted in Figure 5.68. The effect of
increasing slip amplitude on maximum grain diameter when \( N = 10^6 \) cycles for all three materials is plotted in Figure 5.69.

The maximum grain diameter decreases with increasing cycles or with increasing slip amplitude. Specifically, the slope of maximum grain diameter versus the log number of cycles is similar for all three materials. The slope of maximum grain diameter versus slip amplitude is also similar when comparing Ti-5Al-2.5Sn and CP-Ti for any slip amplitude, and at when \( \delta_s \geq 30 \mu m \) for Ti-6Al-4V. The maximum grain diameter is relatively constant when \( \delta_s \geq 60 \mu m \).

5.3.5 Discussion

In this section, the results of mechanical tests and characterization experiments on Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti will be compared and discussed. Of primary interest are damage indicators commonly linked to fretting wear and/or formation of fretting cracks in metals. New information about many of these processes has been discovered due to innovations in characterization tools used in the current work. Orientation maps obtained using EBSD provide one crucial element lacking in previous fretting research, which is the necessary spatial information required to link orientation changes with depth from the fretted surface.

A crystal plasticity algorithm that accounts for heterogeneity associated with the Ti-6Al-4V microstructure has recently been incorporated into a 2-D finite element model (FEM) (Goh et al., 2001). Information derived from EBSD has the added benefit of being generated in a form that is conducive to incorporation into 2-D computational models such as this one. Therefore, it is now possible to make direct links between experimental results and computational models used for fretting life prediction. If serial

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sectioning is done on specimens, this can be extended to 3-D modeling. A 3-D crystal plasticity algorithm for HCP materials is currently under development at Georgia Tech Mayeur and McDowell, 2003). In addition to model validation, it appears that EBSD may be a useful tool for damage assessment through measures such as the misorientation angle distribution and by observing changes in grain diameter that are believed to accompany plastic strain accumulation associated with failure due to fretting.

Fretting only tests indicate that significant cracks can form very early in a test (i.e., less than \( N = 10^3 \) cycles) without a bulk fatigue load superimposed (figures 5.17 and 5.19) and at a relatively low normal load where \( P/Py = 0.05 \). Glaser and Lawless (2001) also observed formation of shallow surface cracks in titanium fretted at applied slip amplitudes of \( \delta_s = 20, 38, \) or \( 75 \mu m \). The fretting disturbed layer in Ti-6Al-4V has a basal texture at intermediate slip amplitudes and higher number of cycles (e.g., Test 1: \( \delta_s = 60 \mu m, N = 10^5 \) cycles). The depth of the fretting disturbed layer in these specimens, 30-50 \( \mu m \), is approximately the same depth as the observed crack length near the edge of contact. EDX analysis near the cracks showed that oxygen had diffused well into the material surrounding the cracks beneath the oxidized/transitional layer. The depth of oxygen diffusion roughly corresponds to the depth of crack formation in the fretting disturbed layer. There were other tests in which a basal texture developed due to fretting, but significant cracks were not observed (i.e., Test 4: \( \delta_s = 120 \mu m, N = 10^4 \) cycles).

Therefore, it appears that a critical slip amplitude threshold exists in which a combination of mechanisms such as plastic deformation, grain reorientation, and oxygen diffusion occurs due to fretting that make conditions ideal for crack formation. The existence of a critical range of slip amplitudes leading to a greater propensity for crack formation and
lower component life corresponds with the trend previously shown in Figure 2.1. Also, Glaeser and Lawless (2001) observed cracking in every specimen fretted at $\delta_n = 38 \mu m$ at a particular normal load, while only isolated tests conducted at lower or higher slip amplitudes exhibited cracking. The contribution of oxygen diffusion to crack formation may be more easily determined if fretting tests are conducted in an inert environment.

The thickness of the oxidized layer appeared to be somewhat dependent upon the applied slip amplitude for all of the tested materials, but tended to stabilize by $N = 10^4$ cycles. An oxidized layer was not observed when $\delta_n = 15 \mu m$ for any of the tested materials. However, the significant increase in low-angle misorientations obtained from EBSD is an indication that a fretting disturbed layer forms at all slip amplitudes by $N = 10^4$. This is a particularly important point for Ti-5Al-2.5Sn and CP-Ti because both had a fairly strong initial texture. A key observation from this work is that fretting induced microstructure evolution may be detected by documenting changes in the misorientation angle distribution even if more obvious visual indications, such as crack formation, “flow” in the grains, an oxidized layer, or a transitional layer, do not exist. In addition, recall that the hysteresis loops and scars appeared similar for all three materials at a given slip amplitude with successive cycling. This highlights why simply studying scar surfaces may be insufficient to quantify damage due to fretting and further illustrates the utility of EBSD as a damage assessment tool.

The orientation of crystals within the oxidized or transitional layers could not be indexed via EBSD, but were nonetheless located using pattern quality maps. This was a further indication of the severe plastic deformation that had occurred in these layers. Pattern quality maps have been used to validate the size of the plastic zone around fatigue
cracks in a fine grained (i.e., 5 μm) nickel based superalloy (Tucker et al., 2000). Therefore, EBSD may be a useful tool for damage assessment even when significant plastic deformation has occurred.

Evidence of twinning was observed in an EBSD orientation map of a CP-Ti specimen fretted at δs = 60 μm and N = 10^3 cycles (Test 21). This was indicated by the misorientation angle around 63° and indices that are associated with compression twins in CP-Ti (Salem et al., 2003). There may be twinning in Ti-5Al-2.5Sn as well, but the results are less conclusive due to the fact that the misorientation angle is around 90° instead of around 60°. However, the indices are the same as those observed in the CP-Ti specimens. Also, there were multiple locations where other misorientations of this sort within grains occurred systematically in fretted specimens of Ti-5Al-2.5Sn and CP-Ti. This trend was not observed in the as-received specimens of either material, nor was it observed in the Ti-6Al-4V fretted or as-received material. EBSD analysis of LCF specimens in which a known strain has been applied may provide more insight into this phenomenon.

The association between misorientation angle distribution and substructures that form due to deformation has been clearly established for some medium to high stacking fault energy metals using TEM (Hansen et al., 2001; Hughes et al., 1996; Wert et al., 1995, Bay, et al. 1992). Gorynin et al. (1988) and Leguey et al. (2002) both indicate that significant strain localization in the form of mesobands or slip bands occur in polycrystalline titanium subjected to deformation either by tension or by fatigue. Gorynin et al. (1988) further observed that the morphology of the substructure is somewhat similar to that observed in FCC materials once a critical strain threshold had
been achieved. Therefore, it is reasonable to assume that observations and models used
to correlate misorientation distribution and plastic strain developed for FCC materials
may be used for comparative purposes in the current work.

Methods used to correlate misorientation angle distribution and plastic strain
have shown some promise (Hughes et al., 1996; Angeliu et al., 2000; Butler et al., 2002),
but are still under development. In general, there appears to be some disconnect in
determining which range of misorientation angles should be used to calculate the scaling
parameter. The scaling parameter used in either case is the average misorientation angle.
Hughes et al. (1996) use cell type as the defining variable, however, the misorientation
angles separating GNBs in FCC materials can be quite high (i.e. around 65°). Because
this method relies on explicit cell type definitions usually obtained only by TEM, it is not
as amenable for incorporation into damage assessment methods using FEA or with
misorientation data obtainable by EBSD. In other words, it is not possible to directly
compare the method Hughes et al. (1996) used to determine the average misorientation
angle and the method Angeliu et al. (2000) used to determine the average intra-grain
misorientation. The relative frequency of misorientation angles corresponding to
formation of GNBs and IDBs (see Figure 2.19) show that deformation results in an
increase in low-angle misorientations, most likely less than about 10°. Therefore, it may
be assumed that a significant portion of deformation evolution may be captured by
documenting low-angle misorientations. Furthermore, the development of low-angle
misorientations appears to be a feature of deformation in ductile metals subjected to a
wide variety of loading scenarios (i.e., tension, compression, fatigue, or sliding). The
misorientation angle distribution, along with changes in grain diameter obtained using
EBSD are perhaps two measures that can provide direct insight into the level of damage that exists due to fretting.

The stacking fault energy of metals studied by Hughes et al. (1995, 1996) were medium to high and the 304 stainless steel studied by Angeliu et al. (2000) is considered a low stacking fault metal (Rainforth et al., 1992). Since stacking fault energy is tied to mobility of screw dislocations, the applicability of models that correlate misorientation distribution to plastic strain for a given material must be carefully considered. More work is needed in this area to determine the dependence of misorientation distribution on stacking fault energy, crystal structure, and alloying content, to name a few.

The misorientation distribution for angles below 5° was observed to increase by as much as four times or more in Ti-5Al-2.5Sn and CP-Ti as compared to the as-received material. This is especially significant in the CP-Ti, as a fairly high relative frequency of low angle misorientations existed in the virgin specimen. This leads to the conclusion that subgrain formation in near surface grains has occurred. The incidence of misorientations around 25° to 30° in CP-Ti and Ti-5Al-2.5Sn may be an indication that strain localization in the form of slip bands (i.e., GNBs) may have formed as well. Interfragment boundaries within adiabatic shear bands in pure polycrystalline titanium deformed in tension were found to have high-angle misorientations of 30° and over (Gorynin et al., 1988). In Ti-6Al-4V, a high relative frequency of low angle misorientations <5° were observed in all of the fretted specimens, so it is likely that subgrain formation in near surface grains due to fretting has also occurred in Ti-6Al-4V. There was a spike in the relative frequency of misorientation angles around 65°, but misorientations angles between GNB’s are usually much lower. Therefore, it is believed

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that the higher angle misorientations in Ti-6Al-4V may be due to constraints imposed by
the Burgers relationship between the α phase and β phase. The plot of relative frequency
in misorientation angles (Figure 5.67) did not show a spike at 65° in CP-Ti or Ti-5Al-
2.5Sn. Then again, the misorientation angle designating compression twins in CP-Ti is
around 63°. The relative frequency plots are generated using uncorrelated data, meaning
that the misorientations from random grains are sampled. This was determined to be the
best method to collect statistically relevant misorientation data. In this case, it is possible
that CP-Ti or Ti-5Al-2.5Sn could exhibit a high relative frequency of misorientation
angles around 65°, but significant twinning would need to be present. This highlights
one of the trade-offs of using EBSD instead of TEM. The exact substructure cannot
always be determined.

In Ti-6Al-4V, the overall trend in relative frequency of misorientation angle
appeared similar when fretting tests for a wide range of conditions were plotted together.
Also, the trend in misorientation angle distribution for Ti-5Al-2.5Sn and CP-Ti looked
similar when plotted together. These results tend to verify the conclusions drawn by
Hughes et al. (1996) and Hansen et al. (2001) that the statistical probability of the
average of all misorientation angles (not just those less than 10°) may be used to estimate
plastic strain for a given material. In other words, the trend in overall misorientation
angle distribution for each material tends to follow a particular pattern. To check this
hypothesis, one recommendation would be to section LCF specimens, then determine the
relative frequency of misorientation angles via EBSD for a given strain amplitude.
Damage due to fretting is dominated by shear deformation, therefore, a series of large
strain torsional tests, followed by EBSD analysis, may also be useful for developing methods used to predict plastic strain in fretted components.

If plastic strain can be estimated based on misorientations distribution obtained by EBSD, the implications for improved fretting life prediction and damage assessment methods are numerous. For instance, models used for fretting life prediction could be directly validated with experimental tests via plastic strain estimates. This may be possible even without consideration of material heterogeneity. However, models developed using crystal plasticity may be more efficient because misorientation distributions linked to plastic deformation in a given material may be input directly into the microstructure configuration. Calculations could then be performed to determine the effect of certain misorientation distributions on component life.

The maximum grain diameter in Ti-5Al-2.5Sn and CP-Ti did not originally appear to decrease significantly due to an increase in the number of cycles or in slip amplitude when the critical misorientation required to define a grain boundary was defined to be 10°. However, when plotted together with results from Ti-6Al-4V, a surprising trend emerged. The decreasing slope of grain diameter versus the number of cycles is nearly identical in comparison with the other materials. The slope of grain diameter versus the slip amplitude is similar for Ti-5Al-2.5Sn and CP-Ti for each test. The slope observed at higher slip amplitudes is nearly identical for all three materials. This trend is important considering that the number of sampled grains, the number of orientation measurements, and the materials are all different. Therefore, it not likely to be an artifact of data post-processing or EBSD experimental setup.
The Hall-Petch model is used to correlate strain hardening behavior with a change in grain (or subgrain) size. It should be pointed out that neither Ti-6Al-4V or Ti-5Al-2.5Sn exhibit significant strain-hardening due to monotonic plastic deformation and both tend to cyclically soften. CP-Ti tends to monotonically strain harden slightly and cyclically hardens beyond a threshold amplitude, so perhaps the Hall-Petch model is more applicable to CP-Ti. Interestingly, even given the differences in bulk material response, the slope of grain diameter with increasing slip amplitude or number of cycles were nearly identical for all three.

Nanoindentation conducted on Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti indicate that the oxidized/transitional layer is significantly harder than the fretting disturbed layer located directly beneath it. However, for both Ti-6Al-4V and Ti-5Al-2.5Sn, the material in the fretting disturbed layer was observed to be softer than the virgin material. In CP-Ti, the material in the fretting disturbed layer was considerably harder than the virgin material. These observations coincide with the cyclic response of Ti-6Al-4V and Ti-5Al-2.5Sn, which tend to exhibit cyclic softening, while CP-Ti tends to exhibit cyclic hardening. Softening in Ti-6Al-4V extended to as deep as 150 μm, after which it approached the hardness of the virgin material. The depth of softening in Ti-5Al-2.5Sn was observed to be about 45 μm, which was the maximum depth at which the indents were made. It is possible that the extent of softening is even deeper in Ti-5Al-2.5Sn. The depth of hardening in CP-Ti was also about 45 μm, after which it approached the hardness of the virgin material. To summarize, it may be concluded that fretting results in hardening or softening in the fretting disturbed layer due to cyclic plasticity that
corresponds with the bulk material response observed in LCF tests for each respective material.

Another consideration in the development of fretting resistant materials and components is the effect of texture on fretting damage. The effect of texture on the frictional behavior of a material is also potentially very important considering the strong relationship between friction coefficient and predicted damage due to fretting (Swalla and Neu, 2000). There is some evidence to suggest that a preferential texture develops in titanium and other hexagonal metals due to sliding (Scott and Wilman, 1958; Farhat, 2001). It has been reported that sliding tends to produce an alignment of basal slip planes (0001) parallel to the worn surface, with the basal plane poles rotated approximately 10° towards the direction of rolling. This trend was certainly observed in the Ti-6Al-4V specimens when δ_h = 60 μm and N = 10^7 cycles or δ_h = 120 μm and N = 10^4 cycles, and to a lesser degree, in the Ti-5Al-2.5Sn specimens as well. The CP-Ti specimens did not exhibit any significant changes in texture.

The initial texture of the Ti-5Al-2.5Sn specimen was very strong, with the [0001] poles oriented in a group centered at about 45° along the TD direction. The hysteresis loops observed in the first 10 cycles in tests conducted at δ_h = 60 μm show considerably more gross-slip in the Ti-5Al-2.5Sn specimens compared to the Ti-6Al-4V specimens. Also, the initial coefficient of friction was much lower in Ti-5Al-2.5Sn compared to Ti-6Al-4V. It is believed that the initial random texture of Ti-6Al-4V results in a higher initial coefficient of friction and less gross slip compared to Ti-5Al-2.5Sn. Once an oxidized layer had formed, however, the hysteresis loops appeared very similar. The oxide layer was observed to form within 1000 cycles in tests conducted at δ_h ≥ 60 μm.
CP-Ti also had a strong initial texture with poles oriented similarly to those observed in the as-received Ti-5Al-2.5Sn bar. Yet, the initial coefficient of friction when $\delta_h = 60 \mu m$ was more than double the initial coefficient of friction observed in the Ti-5Al-2.5Sn. Recall that the CP-Ti grains are very large and so approach the scale of the contact half-width. It is feasible that fretting damage in these CP-Ti specimens are isolated to just a grain or two, therefore, compatibility constraints imposed by the HCP crystal structure may make deformation more difficult. Recall that twinning was observed in near surface grains in CP-Ti when $\delta_h = 60 \mu m$ and $N = 1000$ cycles. A key feature of crystal plasticity modeling is the ability to describe the collective effect of dislocation glide along specific crystallographic slip planes, so this observation may be tested using the 3-D crystal plasticity model that is currently under development.

The texture in the CP-Ti fretting specimens was not significantly different than the texture observed in the as-received specimens. However, the distribution of the poles tended to become more dispersed, generally along the transverse (TD) axis for most fretting tests. Because the CP-Ti grains are large compared to the contact half-width, it is possible that the deformation due to fretting can be accommodated within a few grains, making texture development less likely. Texture development occurs only with successive rotation and sliding of groups of grains. In addition, CP-Ti is considerably more ductile than either Ti-6Al-4V or Ti-5Al-2.5Sn and tends to twin, which makes twinning and substructure formation more probable mechanisms for accommodating plastic strain than grain rotation or sliding. Should any grain rotation or sliding occur, it is most likely occurring along prism planes. In particular, the grains may be simply sliding to-and-fro along prism planes with successive cycling, with the basal plane.
normal direction remaining unchanged. Gorynin et al. (1988) concluded that twinning mechanisms in CP-Ti uniaxial tension specimens are exhausted after the true strain reaches about 0.4, after which the \(<c+a>\) slip mode is activated. The \(<c+a>\) slip mode is along pyramidal planes, which have consistently been found to have a higher critical resolved shear stress than either the prism or the basal planes (which are \(<a>\) type slip modes). Then again, the \(<c+a>\) slip mode (in addition to twinning) is only required to accommodate deformation along the c-axis (refer to Figure 2.13). The large CP-Ti grain size in this case most likely makes deformation along the c-axis less necessary in order to accommodate the plastic strain, so pyramidal slip should not be significant.

There was some texture development observed in the Ti-5Al-2.5Sn specimens, most noticeably when \(\delta_s = 60 \, \mu m\) and \(N = 10^5\) cycles. The pole orientation resembles the basal texture that results from rolling, as defined by Peters et al. (1984). In order to allow grain reorientation of this magnitude, it is likely that slip along prism, basal, and pyramidal planes are all active at higher slip amplitudes and number of cycles. The 3-D crystal plasticity model currently being developed can account for slip on any of these planes, not just along prism planes, which was a primary assumption of the 2-D crystal plasticity model.

A strong pole intensity located along the rolling X(RD) axis was observed in Ti-5Al-2.5Sn at \(\delta_s = 60 \, \mu m\) and \(N = 10^4\) cycles, albeit the overall distribution tends to lie along the transverse (TD) direction. The initial grain size of Ti-5Al-2.5Sn was larger than Ti-6Al-4V, yet still much smaller than the theoretical fretting contact half-width. Therefore, texture development may be more likely in Ti-5Al-2.5Sn compared to CP-Ti simply due to grain size. Because texture development plays such an important role in
the evolution of friction, it is recommended that the effect of grain size on texture development be studied.

The texture results in Ti-6Al-4V indicate that a noticeable reorientation of grains very near the surface within 30-50 \( \mu \text{m} \) occurs due to fretting. A scattering of poles in a loose band rotated at an angle of around 45° relative to the Z(ND) direction was observed for many of the fretting tests as well as poles of medium intensity located directly parallel to the Z(ND) direction. Like the Ti-5Al-2.5Sn specimens however, a very strong basal texture was only observed at the highest number of cycles and when the slip amplitude was of a magnitude that induced gross sliding contact conditions. Interestingly, a strong basal texture and significant oxygen diffusion were observed in the specimen that exhibited the most cracking. Bache et al. (1998) showed that slight variations in the crystallographic orientation of a near \( \alpha \)-Ti alloy can greatly affect the fatigue crack propagation characteristics. Specifically, colonies of \( \alpha \) grains with favorably oriented basal planes exhibited rapid crack development through quasi cleavage facet formation. This hypothesis is supported in part by EBSD analysis of grains surrounding the primary crack in Ti-6Al-4V Test 1 (\( \delta = 60 \mu \text{m} \), \( N = 10^5 \) cycles), where the crack region exhibiting a dramatic change in crack direction corresponds to preferential basal plane orientation. However, other grains surrounding the crack are not oriented accordingly, so it is still not clear if fretting cracks tend grow along preferentially oriented basal planes.

Cracking was not observed in either Ti-5Al-2.5Sn or in CP-Ti. This is especially important in the case of Ti-5Al-2.5Sn, since it has similar mechanical properties and bulk stress/strain behavior as Ti-6Al-4V. These titanium alloys tend to cyclically soften and are considered to be nearly elastic-perfectly plastic (see Figure 5.1). A combination of
factors such as initial grain size and initial texture are likely to play a role in crack formation. The oxidized/transitional layer in Ti-5Al-2.5Sn was thicker and harder than the oxidized layer observed in Ti-6Al-4V, so this layer may have protected or shielded the underlying fretting disturbed layer. In addition, oxygen was not observed to diffuse into the fretting disturbed layer in any of the Ti-5Al-2.5Sn or CP-Ti specimens. It is believed that oxygen diffusion plays an important role in enhancing crack formation in Ti-6Al-4V. Perhaps the interface between α+β lamellae in Ti-6Al-4V acts as a path for oxygen diffusion in the fretting disturbed layer. An EDX microprobe dot pattern in fretted Ti-6Al-4V appeared to indicate oxygen diffusion along α+β lamellae. This may explain why considerable oxygen diffusion may have occurred in the Ti-6Al-4V specimen even in locations relatively far away (i.e. 50 μm) where no visible cracks were observed (see Figures 5.20, 5.21, and 5.22).
Table 5.1: Mechanical properties obtained from monotonic tests.

<table>
<thead>
<tr>
<th>Material</th>
<th>Specimen</th>
<th>σu (MPa)</th>
<th>E (GPa)</th>
<th>Strain rate (s⁻¹)</th>
<th>ν</th>
<th>σv (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>Eylon, 1998</td>
<td>930</td>
<td>118</td>
<td>Not reported</td>
<td>0.349</td>
<td>978</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>AR-421</td>
<td>185</td>
<td>105</td>
<td>5e-3</td>
<td>0.34*</td>
<td>322</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>AR-411</td>
<td>170</td>
<td>98</td>
<td>5e-5</td>
<td>0.34*</td>
<td>309</td>
</tr>
<tr>
<td>Ti-5Al-2.5 Sn</td>
<td>LTS-2</td>
<td>902</td>
<td>122</td>
<td>5e-3</td>
<td>0.33*</td>
<td>927</td>
</tr>
<tr>
<td>Ti-5Al-2.5 Sn</td>
<td>LTS-1</td>
<td>831</td>
<td>123</td>
<td>5e-5</td>
<td>0.33*</td>
<td>854</td>
</tr>
</tbody>
</table>

* obtained from Efunda (www.Efunda.com)

Table 5.2: Plastic constants (monotonic).

<table>
<thead>
<tr>
<th>Material</th>
<th>Specimen</th>
<th>n</th>
<th>H (MPa)</th>
<th>Strain rate (s⁻¹)</th>
<th>m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>Gallagher, 1990</td>
<td>0.008</td>
<td>817</td>
<td>3.6e-3</td>
<td>63</td>
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<tr>
<td>CP-Ti</td>
<td>AR-421</td>
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<td>400</td>
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<td>.53</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>AR-411</td>
<td>0.136</td>
<td>394</td>
<td>5e-5</td>
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</tr>
<tr>
<td>Ti-5Al-2.5 Sn</td>
<td>LTS-2</td>
<td>0.008</td>
<td>958</td>
<td>5e-3</td>
<td>56</td>
</tr>
<tr>
<td>Ti-5Al-2.5 Sn</td>
<td>LTS-1</td>
<td>0.016</td>
<td>895</td>
<td>5e-5</td>
<td></td>
</tr>
</tbody>
</table>

Table 5.3: Fatigue constants for Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti.

<table>
<thead>
<tr>
<th>Material</th>
<th>σu' (MPa)</th>
<th>b</th>
<th>εu'</th>
<th>c</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>2572</td>
<td>-0.14</td>
<td>319.0</td>
<td>-1.51</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>1525</td>
<td>-0.09</td>
<td>80.2</td>
<td>-1.35</td>
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<tr>
<td>CP-Ti</td>
<td>357</td>
<td>-0.06</td>
<td>0.053</td>
<td>-0.31</td>
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</tbody>
</table>

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Table 5.4: Strain life data for Ti-5Al-2.5Sn.

<table>
<thead>
<tr>
<th>Test</th>
<th>$\varepsilon_a$ (MPa)</th>
<th>$\sigma_a$ (MPa)</th>
<th>$R$</th>
<th>Strain rate (1/s) 126</th>
<th>E (GPa) 775</th>
<th>$\sigma_{\text{max}}$ (MPa) -646</th>
<th>$\sigma_{\text{min}}$ (MPa) 4.92E-3</th>
<th>$\varepsilon_a$ (elastic) 8.09E-5</th>
<th>$\varepsilon_a$ (plastic) 9660</th>
</tr>
</thead>
<tbody>
<tr>
<td>L15-3</td>
<td>5.00E-3</td>
<td>620</td>
<td>-1</td>
<td>5.00E-03</td>
<td>126</td>
<td>775</td>
<td>-646</td>
<td>4.92E-3</td>
<td>8.09E-5</td>
</tr>
<tr>
<td>L15-4</td>
<td>7.50E-3</td>
<td>701</td>
<td>-1</td>
<td>5.00E-03</td>
<td>114</td>
<td>704</td>
<td>-698</td>
<td>6.14E-3</td>
<td>1.36E-3</td>
</tr>
<tr>
<td>L15-5</td>
<td>1.00E-2</td>
<td>811</td>
<td>-1</td>
<td>5.00E-03</td>
<td>125</td>
<td>790</td>
<td>-831</td>
<td>6.50E-3</td>
<td>3.50E-3</td>
</tr>
<tr>
<td>L15-5</td>
<td>1.38E-2</td>
<td>769</td>
<td>-1</td>
<td>5.00E-03</td>
<td>117</td>
<td>755</td>
<td>-784</td>
<td>6.56E-3</td>
<td>7.19E-3</td>
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Table 5.5: Strain-life data for CP-Ti.

<table>
<thead>
<tr>
<th>Test</th>
<th>$\varepsilon_a$ (MPa)</th>
<th>$\sigma_a$ (MPa)</th>
<th>$R$</th>
<th>Strain rate (1/s) 113</th>
<th>E (GPa) 165</th>
<th>$\sigma_{\text{max}}$ (MPa) -159</th>
<th>$\sigma_{\text{min}}$ (MPa) 1.44E-3</th>
<th>$\varepsilon_a$ (elastic) 1.06E-3</th>
<th>$\varepsilon_a$ (plastic) 10513</th>
</tr>
</thead>
<tbody>
<tr>
<td>L211</td>
<td>2.50E-3</td>
<td>162</td>
<td>-1</td>
<td>5.00E-03</td>
<td>113</td>
<td>165</td>
<td>-159</td>
<td>1.44E-3</td>
<td>1.06E-3</td>
</tr>
<tr>
<td>L212</td>
<td>5.00E-3</td>
<td>217</td>
<td>-1</td>
<td>5.00E-03</td>
<td>112</td>
<td>216</td>
<td>-219</td>
<td>1.95E-3</td>
<td>3.05E-3</td>
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<tr>
<td>L311</td>
<td>8.00E-2</td>
<td>286</td>
<td>-1</td>
<td>5.00E-03</td>
<td>114</td>
<td>294</td>
<td>-276</td>
<td>2.51E-3</td>
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<td>L221</td>
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<td>246</td>
<td>-1</td>
<td>5.00E-03</td>
<td>108</td>
<td>254</td>
<td>-238</td>
<td>2.29E-3</td>
<td>1.15E-3</td>
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Table 5.6: Comparison of theoretical scar width and measured width of fretting scars after experiments.

<table>
<thead>
<tr>
<th>Test</th>
<th>Specimen number</th>
<th>Nominal slip amp (µm)</th>
<th>Cycles</th>
<th>Theoretical scar width (µm)</th>
<th>Measured scar width (µm)</th>
<th>Actual theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>2</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>3</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>4</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>5</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>6</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>7</td>
<td>113-S912</td>
<td>703 60</td>
<td>10000</td>
<td>410.9</td>
<td>1200.0</td>
<td>2.8</td>
</tr>
<tr>
<td>21</td>
<td>AR-S131</td>
<td>294 60</td>
<td>1000</td>
<td>314.0</td>
<td>180.0</td>
<td>5.1</td>
</tr>
<tr>
<td>22</td>
<td>AR-S131</td>
<td>294 60</td>
<td>1000</td>
<td>314.0</td>
<td>180.0</td>
<td>5.1</td>
</tr>
<tr>
<td>23</td>
<td>AR-S131</td>
<td>294 60</td>
<td>1000</td>
<td>314.0</td>
<td>180.0</td>
<td>5.1</td>
</tr>
<tr>
<td>24</td>
<td>AR-S131</td>
<td>294 60</td>
<td>1000</td>
<td>314.0</td>
<td>180.0</td>
<td>5.1</td>
</tr>
<tr>
<td>25</td>
<td>AR-S131</td>
<td>294 60</td>
<td>1000</td>
<td>314.0</td>
<td>180.0</td>
<td>5.1</td>
</tr>
<tr>
<td>12</td>
<td>STS_1</td>
<td>640 120</td>
<td>10000</td>
<td>516.2</td>
<td>2000.0</td>
<td>3.9</td>
</tr>
<tr>
<td>14</td>
<td>STS_1</td>
<td>640 120</td>
<td>10000</td>
<td>396.2</td>
<td>1100.0</td>
<td>2.8</td>
</tr>
<tr>
<td>15</td>
<td>STS_1</td>
<td>640 120</td>
<td>10000</td>
<td>396.2</td>
<td>1100.0</td>
<td>2.8</td>
</tr>
<tr>
<td>16</td>
<td>STS_1</td>
<td>640 120</td>
<td>10000</td>
<td>396.2</td>
<td>1100.0</td>
<td>2.8</td>
</tr>
<tr>
<td>17</td>
<td>STS_1</td>
<td>640 120</td>
<td>10000</td>
<td>516.2</td>
<td>3700.0</td>
<td>7.2</td>
</tr>
<tr>
<td>18</td>
<td>STS_1</td>
<td>640 120</td>
<td>10000</td>
<td>396.2</td>
<td>1800.0</td>
<td>4.6</td>
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Table 5.7: Coefficient of friction comparison (cycle 10).

<table>
<thead>
<tr>
<th>Material</th>
<th>μ</th>
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<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>0.71</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>0.51</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>1.4</td>
</tr>
</tbody>
</table>

Table 5.8: Average hardness of indents in fretted Ti-6Al-4V (Test 1) specimen and in virgin Ti-6Al-4V plate.

<table>
<thead>
<tr>
<th>Row number</th>
<th>Average hardness in specimen (GPa)</th>
<th>Average hardness in virgin material (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>3.44</td>
<td>4.39</td>
</tr>
<tr>
<td>3</td>
<td>4.15</td>
<td>4.33</td>
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<tr>
<td>4</td>
<td>3.96</td>
<td>4.23</td>
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<tr>
<td>5</td>
<td>3.94</td>
<td>4.59</td>
</tr>
<tr>
<td>6</td>
<td>4.08</td>
<td>4.33</td>
</tr>
<tr>
<td>7</td>
<td>4.07</td>
<td>4.11</td>
</tr>
<tr>
<td>8</td>
<td>4.59</td>
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<td>9</td>
<td>4.64</td>
<td>4.40</td>
</tr>
<tr>
<td>10</td>
<td>4.23</td>
<td>4.35</td>
</tr>
</tbody>
</table>

Table 5.9: Summary of texture analysis results shown in Figure 5.29.

<table>
<thead>
<tr>
<th>N (cycles)</th>
<th>D_{max} (μm)</th>
<th>N_p</th>
<th>N_o</th>
<th>Angle (°)/direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>10⁷</td>
<td>32</td>
<td>94</td>
<td>817</td>
<td>75°/RD</td>
</tr>
<tr>
<td>10³</td>
<td>18</td>
<td>105</td>
<td>3919</td>
<td>70°/RD</td>
</tr>
<tr>
<td>10⁵</td>
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<td>37</td>
<td>2768</td>
<td>80°</td>
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</table>

Table 5.10: Summary of texture analysis results shown in Figure 5.32.

<table>
<thead>
<tr>
<th>δ_p (μm)</th>
<th>D_{max} (μm)</th>
<th>N_p</th>
<th>N_o</th>
<th>Angle (°)/direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>16</td>
<td>163</td>
<td>5500</td>
<td>55°</td>
</tr>
<tr>
<td>60</td>
<td>18</td>
<td>105</td>
<td>3919</td>
<td>65°</td>
</tr>
<tr>
<td>120</td>
<td>22</td>
<td>47</td>
<td>1434</td>
<td>0°</td>
</tr>
</tbody>
</table>

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Table 5.11: Average hardness of indents in Ti-5Al-2.5Sn specimen and in virgin Ti-5Al-2.5Sn bar.

<table>
<thead>
<tr>
<th>Row number</th>
<th>Average hardness in specimen (GPa)</th>
<th>Average hardness in virgin material (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>7.63</td>
<td>4.07</td>
</tr>
<tr>
<td>3</td>
<td>3.57</td>
<td>3.79</td>
</tr>
<tr>
<td>4</td>
<td>3.84</td>
<td>4.14</td>
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</table>

Table 5.12: Summary of texture analysis results shown in Figure 5.47.

<table>
<thead>
<tr>
<th>N (cycles)</th>
<th>D_{max} (μm)</th>
<th>N_e</th>
<th>N_o</th>
<th>Angle (θ)/direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>$10^3$</td>
<td>45</td>
<td>117</td>
<td>8559</td>
<td>20°/RD 65°</td>
</tr>
<tr>
<td>$10^3$</td>
<td>41</td>
<td>156</td>
<td>8932</td>
<td>40° 35°</td>
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</table>

Table 5.13: Summary of texture analysis results shown in Figure 5.48.

<table>
<thead>
<tr>
<th>δ_n (μm)</th>
<th>D_{max} (μm)</th>
<th>N_e</th>
<th>N_o</th>
<th>Angle (θ)/direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>60</td>
<td>133</td>
<td>9360</td>
<td>70°/TD 40°/TD</td>
</tr>
<tr>
<td>30</td>
<td>47</td>
<td>159</td>
<td>10235</td>
<td>Distributed along TD axis</td>
</tr>
<tr>
<td>60</td>
<td>45</td>
<td>117</td>
<td>8559</td>
<td>20°/RD 30°/RD 65°</td>
</tr>
<tr>
<td>120</td>
<td>46</td>
<td>154</td>
<td>4362</td>
<td>20°/RD 75°/TD</td>
</tr>
</tbody>
</table>

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Table 5.14: Average hardness of indents in fretted CP-Ti specimen and in virgin CP-Ti plate.

<table>
<thead>
<tr>
<th>Row number</th>
<th>Average hardness in fretted specimen (GPa)</th>
<th>Average hardness in virgin material (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.84</td>
<td>1.34</td>
</tr>
<tr>
<td>2</td>
<td>1.18</td>
<td>1.08</td>
</tr>
<tr>
<td>3</td>
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</table>

Table 5.15: Summary of texture analysis results shown in Figure 5.63.

<table>
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<tr>
<th>N cycles</th>
<th>$D_{max}$ (μm)</th>
<th>$N_{g}$</th>
<th>$N_{a}$</th>
<th>Angle (θ)/direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>$10^3$</td>
<td>163</td>
<td>99</td>
<td>5923</td>
<td>45°/TD 75°</td>
</tr>
<tr>
<td>$10^4$</td>
<td>142</td>
<td>236</td>
<td>5689</td>
<td>70°/TD</td>
</tr>
</tbody>
</table>

Table 5.16: Summary of texture analysis results shown in Figure 5.64.

<table>
<thead>
<tr>
<th>$\delta_s$ (μm)</th>
<th>$D_{max}$ (μm)</th>
<th>$N_{g}$</th>
<th>$N_{a}$</th>
<th>Angle (θ)/direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>156</td>
<td>54</td>
<td>12997</td>
<td>65°/TD</td>
</tr>
<tr>
<td>30</td>
<td>142</td>
<td>61</td>
<td>3692</td>
<td>55°/TD</td>
</tr>
<tr>
<td>60</td>
<td>142</td>
<td>236</td>
<td>5689</td>
<td>70°/TD</td>
</tr>
<tr>
<td>120</td>
<td>136</td>
<td>221</td>
<td>1237</td>
<td>60°/TD</td>
</tr>
</tbody>
</table>
Figure 5.1: Cyclic stress-strain curve of Ti-6Al-4V in elastic regime (Dunyak, 1999).

Figure 5.2: Initial reversal and cyclic stress-strain curve for Ti-6Al-4V (Kurath, 1999).
Figure 5.3: Stabilized (half-life) cyclic stress-strain curve for Ti-6Al-4V (Kurath, 1999).
Figure 5.4: Monotonic and cyclic stress-strain curves for CP-Ti, Ti-5Al-2.5Sn, and Ti-6Al-4V.

Figure 5.5: Ti-6Al-4V strain-life curves.

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Figure 5.6: Ti-5Al-2.5Sn strain-life curves.

Figure 5.7: CP-Ti strain-life curves.
Figure 5.8: Comparison of strain life curves for Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti.

![Graph showing strain life curves for different materials.]

Figure 5.9: Frictional force versus clip gage displacement hysteresis plots at $N = 10^3$ cycles for fretting tests on Ti-6Al-4V.

(a) $\delta_s = 15 \mu m$  
(b) $\delta_s = 30 \mu m$  
(c) $\delta_s = 60 \mu m$  
(d) $\delta_s = 120 \mu m$
Figure 5.10: Evolution of the frictional force per pad versus clip gage displacement hysteresis during a fretting experiment on Ti-6Al-4V; Test 3, \( \delta_n = 60 \mu m, N = 10^6 \) cycles.
Figure 5.11: Frictional force range versus cycles for a fretting experiment on Ti-6Al-4V; Test 3, $\delta_s = 60 \mu m$, $N = 10^4$ cycles.

Figure 5.12: Clip gage displacement amplitude versus cycles for a fretting test on Ti-6Al-4V; Test 3, $\delta_s = 60 \mu m$, $N = 10^4$ cycles.
Figure 5.13: Evolution of the frictional force per pad versus clip gage displacement hysteresis during a fretting experiment on Ti-5Al-2.5Sn; Test 14, $\delta_a = 60 \mu m$, $N = 10^4$ cycles.
Figure 5.14: Evolution of the frictional force per pad versus clip gage displacement hysteresis during a fretting experiment on CP-Ti; Test 22, $\delta_s = 60$ $\mu$m, $N = 10^5$ cycles.
Figure 5.15: Schematic showing fretting scar orientation relative to specimen and plate.

Figure 5.16: Scanned image of Ti-6Al-4V fretting scars on both sides of specimen.
Figure 5.17: SEM micrograph of Ti-6Al-4V fretting scar cross section etched with 0.5% HF; Test 2 ($\delta_0 = 60$ $\mu$m, $N=10^6$), (a) near edge of fretting contact (b) near center of fretting contact.
Figure 5.18: (a) SEM micrograph of Ti-6Al-4V (Test 2, $R_n = 60 \mu m$, N=10^7 cycles) and (b) EDS line scan illustrating change of composition with depth along line shown in image.
Figure 5.19: SEM micrograph of Ti-6Al-4V fretting scar cross section etched with 0.5% HF; Test 1 ($d_s = 60 \mu m$, $N=10^9$ cycles).
Figure 5.20: (a) SEM micrograph of Ti-6Al-4V (Test 1, \( \delta_s = 60 \, \mu\text{m}, N=10^9 \) cycles) and (b) EDS line scan illustrating change of composition with depth along line shown in image.
Figure 5.21: (a) SEM micrograph of Ti-6Al-4V (Test 1, \( \delta_t = 60 \text{ \( \mu \)m}, N=10^5 \text{ cycles} \)) and (b) EDS line scan illustrating change of composition with depth along line shown in image.
Figure 5.22: (a) SEM micrograph of Ti-6Al-4V (Test 1, δs = 60 μm, N=10⁵ cycles) and (b) EDS line scan illustrating change of composition with depth along line shown in image.
Figure 5.23: Nanoindentation test results on Ti-6Al-4V (a) Test 1 ($\delta_s = 60 \, \mu m$, $N=10^3$), and (b) Test 2 ($\delta_s = 60 \, \mu m$, $N=1000$).
Figure 5.24: Orientation map legend.

Figure 5.25: (a) Orientation map, (b) pattern quality map, and (c) and inverse pole plot for Ti-6Al-4V fretting specimen (Test 1, $\delta_s = 60\, \mu m$, $N = 10^5$ cycles).
Figure 5.26: (a) SEM micrograph, (b) orientation map, (c) pattern quality map and (d) an inverse pole plot of Ti-6Al-4V fretting specimen; Test 2 (δ_a = 60 μm, N =10^5 cycles).
Figure 5.27: Pole figure showing texture in longitudinal cross section of as-received Ti-6Al-4V plate, $D_{max} = 38 \, \mu m, N_e = 143$.

Figure 5.28: Misorientation distributions from cross-sections is the as-received Ti-6Al-4V plate for the longitudinal (RD) direction.
Figure 5.29: Pole figures showing effect of cycles on texture for Ti-6Al-4V specimens within 30-50 μm in the fretting disturbed layer when δₜ = 60 μm, (a) 10³ cycles, (b) 10⁴ cycles, and (c) 10⁵ cycles.
Figure 5.30: (a) Texture component map and (b) inverse pole plot showing grains with basal plane normal oriented within 45° from Z(ND) for Ti-6Al-4V fretting Test 1: (δ = 60 μm, N = 10^6 cycles).
Figure 5.31: Misorientation distributions for Ti-6Al-4V specimens within 30-50 μm in the fretting disturbed layer when δ = 60 μm and (a) 10^5 cycles, (b) 10^6 cycles, and (c) 10^7 cycles.
Figure 5.32: Pole figures showing effect of slip amplitude on Ti-6Al-4V texture within 30-50 μm in fretting disturbed region when N = 10^4 cycles (a) δ_s = 15 μm, (b) δ_s = 30 μm, (c) δ_s = 60 μm, and (d) δ_s = 120 μm.
Figure 5.33: (a) Texture component map and (b) inverse pole plot showing grains with basal plane normal oriented within 45° from Z(ND) for Ti-6Al-4V fretting Test 4: ($d_s = 120 \, \mu m$, $N = 10^6$ cycles).
Figure 5.34: Misorientation distributions for Ti-6Al-4V specimens within 30-50 μm in the fretting disturbed layer when N =10⁴ and (a) δₜ = 15 μm, (b) δₜ = 30 μm, (c) δₜ = 60 μm, (d) δₜ = 120 μm.
Figure 5.35: (a) SEM micrograph and (b) texture component map with inverse pole plot illustrating orientation of grains around primary crack in Ti-6Al-4V; Test 1 (δ = 60 μm, N = 10⁵ cycles).
Figure 5.36: Schematic showing fretting specimen and fretting scar orientation relative to Ti-5Al-2.5Sn bar.

Figure 5.37: Scanned image of Ti-5Al-2.5Sn fretting scars on both sides of specimen.
Figure 5.38: SEM micrographs of Ti-5Al-2.5Sn fretting scar cross section etched with 0.5% HF; Test 12 ($d_a = 120 \, \mu m$, $N=10^4$ cycles).
Figure 5.39: SEM micrograph of Ti-5Al-2.5Sn fretting scar cross section etched with 0.5% HF; Test 14 ($\delta_c = 60 \mu m$, $N=10^4$ cycles).
Figure 5.40: (a) SEM micrograph of Ti-5Al-2.5Sn; Test 12 (δ₀ = 120 μm, N=10⁶ cycles) and (b) EDS line scan illustrating change of composition with depth along line shown in image.
Figure 5.41: (a) SEM micrograph of Ti-5Al-2.5Sn : Test 14 (δₐ = 60 μm, N=10⁶ cycles) and (b) EDS line scan illustrating change of composition with depth along line shown in image.
Figure 5.42: (a) SEM micrograph (b) orientation map, (c) pattern quality map and (d) inverse pole plot of Ti-5Al-2.5Sn fretting specimen; Test 18 (δk = 60 μm, N = 10⁶ cycles).
Figure 5.43: (a) orientation map, (b) inverse pole plot, and (c) pattern quality map of Ti-5Al-2.5Sn fretting specimen; Test 16 (δ = 15 μm, N = 10^6 cycles).
Figure 5.44: Nanindentation test results on Ti-5Al-2.5Sn Test 12 ($\delta_n = 120 \mu m$, N=10$^4$ cycles).
Figure 5.45: Pole figure showing texture in longitudinal cross section of as-received Ti-5Al-2.5Sn bar. $D_{\text{max}} = 70 \mu\text{m}$, $N_g = 177$.

Figure 5.46: Misorientation distribution for longitudinal cross section of as-received Ti-5Al-2.5Sn bar.
Figure 5.47: Pole figures illustrating effect of cycles on texture for Ti-5Al-2.5Sn specimens within 50-70 μm in fretting disturbed layer when δₖ = 60 μm and (a) 10⁶ cycles, (b) 10⁸ cycles.

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Figure 5.61: Pole figure showing texture in longitudinal cross section of as-received CP-Ti plate. $D_{max} = 206 \, \mu m$, $D_{avg} = 42 \, \mu m$, $N_t = 217$. 

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Figure 5.64: Pole figures illustrating effect of slip amplitude on texture for CP-Ti specimens within 200-220 μm in fretting disturbed layer in tests with 10^6 cycles, (a) $\delta_s = 15 \mu m$, (b) $\delta_s = 30 \mu m$, (c) $\delta_s = 60 \mu m$, (d) $\delta_s = 120 \mu m$. 

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CHAPTER VI
CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

An experimental program was undertaken to shed light on deformation mechanisms that lead to fretting crack formation at the microstructural level in industry relevant α+ β Ti-6Al-4V. In addition, CP-Ti and Ti-5Al-2.5Sn were studied to determine the role of the α-phase in fretting damage as well as offer more insight into HCP material behavior.

Nearest the surface, a layer forms that appears porous and brittle and is often filled with microcracks and voids. It is considered to be an oxidized layer because the concentration of oxygen is highest in this area. Directly beneath the oxidized layer, an intermediate or transitional layer exists that is characterized by severe plastic deformation as indicated by pile up and layering of material. The precise morphology of this region is not clear. In the current study, the oxygen concentration here is observed to be much less than in the oxidized region above it, but is still relatively high, hence the term “transitional” layer. A transitional layer is not always visually observed or is only observed in pockets.

The microstructure in the next layer appears similar to the virgin microstructure, though grains have been distorted, likely due to large plastic deformation. This region, termed a fretting disturbed layer is often visually distinguished by flow or rotation of
grains and/or formation of primary cracks growing into the specimen. The concentration of oxygen found in this region often, but not always, drops to a level that is not much higher than the concentration found in the virgin material. The bulk material region is the area in which no visible evidence of plastic deformation is observed, nor is there any evidence of an increase in oxygen concentration in this region. The bulk material is assumed to have the same mechanical properties and composition as the virgin material.

Fretting only tests indicate that significant cracks can form in Ti-6Al-4V very early in a test (i.e. less than $10^5$ cycles) without a bulk fatigue load superimposed and at a relatively low normalized normal load of $F/F_y = 0.05$. EDX analysis near the cracks showed that oxygen has diffused well into the material surrounding the cracks beneath the oxidized/transitional layer. The depth of oxygen diffusion roughly corresponds to the depth of crack formation in the fretting disturbed layer. The interface between $\alpha$+$\beta$ lamellae in Ti-6Al-4V may act as a path for oxygen diffusion in the fretting disturbed layer, which may partially explain why significant cracking was not observed in the Ti-5Al-2.5Sn specimens even though the bulk material properties for these two titanium alloys are similar.

A strong basal microtexture and significant oxygen diffusion were observed in the Ti-6Al-4V specimen that exhibited the most significant cracking. However, a Ti-6Al-4V specimen tested at a higher slip amplitude also exhibited a strong basal microtexture, but neither oxygen diffusion nor cracking were observed. Therefore, it appears that a critical slip amplitude threshold exists in which a combination of mechanisms such as plastic deformation, grain reorientation, and oxygen diffusion occurs due to fretting that make conditions ideal for crack formation.
A strong basal microtexture in the fretting disturbed layer was observed in Ti-6Al-4V and Ti-5Al-2.5Sn only at intermediate to high slip amplitudes and a higher number of cycles. The CP-Ti texture after fretting was not observed to deviate much from the initial texture, which is attributed in part to the fact that the initial grain size is of the same order of magnitude as the contact half-width. It is feasible that fretting damage in these CP-Ti specimens are isolated to just a grain or two because the initial grain size is of the same magnitude as the contact half-width. In this case, the plastic strain may be accommodated by twinning and/or subgrain formation instead of planar glide. Also, compatibility constraints imposed by the HCP crystal structure may make deformation more difficult. This hypothesis may be confirmed by evidence of twinning observed in near surface grains in CP-Ti when $\delta_s = 60 \, \mu m$ and $N = 1000$ cycles.

In order for microtexture development to occur, the grains within the fretting disturbed layer must experience out-of-plane rotation which can only be activated by deformation in the c-direction of the HCP unit cell. Deformation in the c-direction is only accomplished by slip along the pyramidal plane or by twinning. Since the pyramidal slip plane has the highest critical resolved shear stress compared to either basal slip or prismatic slip, it is more likely that the plastic strain in relatively large grained CP-Ti is accommodated by subgrain formation and twinning with perhaps some prism slip, none of which leads to microtexture development. In Ti-6Al-4V and Ti-5Al-2.5Sn, two materials that do not exhibit twinning, slip more likely occurs primarily along prismatic and basal planes (in that order), and the pyramidal slip mode is only activated at the higher slip amplitudes, thereby allowing re-orientation of the basal planes into a basal microtexture.
The hysteresis loops observed in the first 10 cycles in tests conducted at $\delta_s = 60$ µm show considerably more gross-slip in the Ti-5Al-2.5Sn specimens compared to the Ti-6Al-4V or CP-Ti specimens. Also, the coefficient of friction at the 10th cycle was much lower in Ti-5Al-2.5Sn compared to either material. It is hypothesized that the initial random texture of Ti-6Al-4V results in a higher initial coefficient of friction and less gross slip compared to Ti-5Al-2.5Sn. The frictional force range (as observed in the hysteresis loops) for all Ti-6Al-4V tests were routinely more variable compared to those for Ti-5Al-2.5Sn and CP-Ti, thereby confirming the role that initial texture may play in the early fretting behavior. However, once an oxidized layer has formed, the hysteresis loops appear very similar. The oxide layer was observed to form within 1000 cycles in tests conducted at $\delta_s \geq 60$ µm and by $10^4$ cycles at $\delta_s = 30$µm. The thickness of the oxidized layer in Ti-6Al-4V, Ti-5Al-2.5Sn and CP-Ti appeared to be dependent upon the applied slip amplitude, but tended to stabilize by $N = 10^4$ cycles.

A significant increase in low-angle misorientations (<10°) in the fretting disturbed layer is observed in all tested materials at all slip amplitudes by $N = 10^4$ cycles. A key observation from this work is that fretting induced microstructure evolution may be detected by documenting changes in the misorientation angle distribution even if more obvious visual indications, such as crack formation, “flow” in the grains, an oxidized layer, or a transitional layer, do not exist. The maximum grain size was also observed to decrease at the same rate for all three materials for a given number of cycles $\propto$ slip amplitude. Therefore, the misorientation angle distribution, along with changes in grain diameter obtained using EBSD are perhaps two measures that can provide direct insight into the extent of damage that exists due to fretting.
The orientation of crystals within the oxidized or transitional layers could not be
indexed via EBSD, but were nonetheless located using pattern quality maps. The
absence or diffuseness of EBSD patterns is an indication of the severe plastic
deformation that has occurred in these layers. Therefore, EBSD can be used to detect
damage due to fretting even when the grains cannot be indexed due to extreme plastic
deformation.

Nanoindentation conducted on Ti-6Al-4V, Ti-5Al-2.5Sn, and CP-Ti indicate that
the oxidized/transition layer contains pockets that are significantly harder than the
fretting disturbed layer located directly beneath it. However, for both Ti-6Al-4V and Ti-
5Al-2.5Sn, the material in the fretting disturbed layer was observed to be softer than the
virgin material. In CP-Ti, the material in the fretting disturbed layer was considerably
harder than the virgin material. These observations coincide with the cyclic response of
Ti-6Al-4V and Ti-5Al-2.5Sn, which tend to exhibit cyclic softening, while CP-Ti tends to
exhibit cyclic hardening. Therefore, it may be concluded that fretting results in
hardening or softening in the fretting disturbed layer due to cyclic plasticity that
corresponds with the bulk material response observed in uniaxial LCF tests for each
respective material.

6.2 Recommendations

The following recommendations are made based on results from this study:

- It is not clear which micromechanical mechanisms control the level of oxygen
diffusion observed in the fretting disturbed layer of the Ti-6Al-4V specimen
containing the most cracks. While a superficial oxide layer developed at
intermediate slip amplitudes or higher in all materials, significant cracking was not always observed. The effect of oxygen on layer formation and the contribution of oxygen diffusion to crack formation could be further investigated by conducting fretting tests in an inert environment.

- The medical community uses Ti-6Al-4V for a wide variety of implant components, such as hip replacement joints, that tend to experience fretting related failures. Therefore, some opportunity exists to conduct fretting experiments geared towards the orthopaedic implant industry.

- Computational or empirical methods used to correlate misorientation angle distribution with plastic strain have shown some promise, but are still in the very early stages of development. EBSD analysis of specimens in which a known uniaxial and/or torsional cyclic plastic strain has been applied may provide more insight into the evolution of misorientation angle distribution and its relationship to plastic strain. By calibrating the relative frequency of misorientation angles for a given strain amplitude, direct validation of computational crystal plasticity models and experimental results is also possible.

- It would be useful to perform EBSD analysis on actual components, such as a gas turbine fan blade, to directly determine if the misorientation angle distribution could be used as a damage assessment measure. For example, one study could involve comparison between three blades: Two would receive the same prior surface treatment, such as laser shock peening, etc. However, one blade would be unfielded, i.e. had not been in service, while the second would have suffered some fretting damage in actual service. The third would be a blade...
that had not received any prior surface treatment and was not in service. In this way, the sensitivity of the misorientation angle distribution to prior surface treatments, as well as the ability to distinguish damage due to fretting could be clarified.

- Procedures used to correlate misorientation angle distribution with plastic strain have been investigated for materials such as aluminum, nickel, and stainless steel. Once a method has been developed to correlate the misorientation angle distribution with plastic strain, as mentioned above, there would be a need to determine the dependence of the misorientation angle distribution on stacking fault energy, crystal structure, and alloying content.

- Results show that initial texture and grain size plays a crucial role in texture development and the evolution of friction. In computational analyses used for fretting life prediction, the friction coefficient is used to account for surface effects that are otherwise very difficult to model. In other words, there is a strong relationship between the friction coefficient and predicted damage due to fretting. Since texture development plays such an important role in the evolution of friction, it is recommended that the effect of grain size and initial texture on subsequent texture development be included in future experimental and computational fretting studies.

- Using TEM, precise deformation substructure and related misorientation angle distribution may be directly observed. It is recommended that TEM methods be pursued as soon as software and hardware improvements required to automate orientation mapping become feasible.
APPENDIX A

LOW CYCLE FATIGUE TEST DATA
Low Cycle Fatigue Test Data

Funding Agency: AFOSR
Specimen drawing: per ASTM E606
Machining Source: Cincinnati Testing Labs
(513) 851-3313
Test temp: Room (a70°F)
Control Mode: Axial strain
Operator: Dana Swalla

Waveform: Ramp
Strain rate: 0.005 sec⁻¹
Strain range: 0.5%

Equipment: 20 kip MTS 923.14
Test Date: 1/19/02

Specimen I.D.: AR-L211

Nf = 105,313 (50% load drop)
Failure location: Gage, inside extensometer

R.T. O.D (mm/in): 6.28/0.247
R.T. Area (mm²/in²): 30.9/0.048
Gage length (mm/in): 25.4/1.00

1st Cycle
Young's Modulus: 112.8/16.36
(GPa/ksi)
Δε (%): 0.50
Max σ (MPa/ksi): 173.4/25.14
Min σ (MPa/ksi): -179.1/-25.98

Mid life cycle (52000)
Young's Modulus: 99.25/14.39
(GPa/ksi)
Max σ (MPa/ksi): 165.0/23.93
Min σ (MPa/ksi): -159.0/-23.05
Δε (%): 0.500
Δε (_): 0.287
Δε (_): 0.212
Low Cycle Fatigue Test Data

Funding Agency: AFOSR
Specimen drawing: per ASTM E606
Machining Source: Cincinnati Testing Labs
(313) 851-3313
Control Mode: Axial strain
Operator: Dana Swalla

Waveform: Ramp
Strain rate: 0.005 sec\(^{-1}\)
Test temp: Room (\text{at} 70^\circ\text{F})
Strain range: 1.0%

Equipment: 20 kip MTS 923.14
Test Date: 1/18/02

Specimen I.D.: AR-L.121
Nf = 6209 (50% load drop)
Failure location: Gage, inside extensometer
R.T. O.D (mm/in): 6.32/0.249
R.T. Area (mm\(^2\)/in\(^2\)): 31.4/0.049
Gage length (mm/in): 25.4/1.00

1st Cycle
Young’s Modulus: 111.7/16.19 (GPa/ksi)
\(\Delta e\) (%): 1.01
Max \(\sigma\) (MPa/ksi): 220.90/32.01
Min \(\sigma\) (MPa/ksi): -232.22/-33.66

Mid life cycle (3110)
Young’s Modulus: 84.57/12.26 (GPa/ksi)
Max \(\sigma\) (MPa/ksi): 216.4/31.37
Min \(\sigma\) (MPa/ksi): -219.2/-31.77
\(\Delta e\) (%): 1.0
\(\Delta e_e\) (%): 0.389
\(\Delta e_p\) (%): 0.610
Low Cycle Fatigue Test Data

Funding Agency: AFOSR  Waveform: Ramp
Specimen drawing: per ASTM E606  Strain rate: 0.005 sec⁻¹
Machining Source: Cincinnati Testing Labs  Strain range: 1.6%
(513) 851-3313  Test temp: Room (±70°F)
Control Mode: Axial strain  Equipment: 20 kip MTS 923.14
Operator: Dana Swalla  Test Date: 1/16/02
Specimen I.D.: AR-L311

Nf = 1023 (50% load drop)
Failure location: Gage, inside extensometer
R.T. O.D (mm/in): 6.34/0.2497
R.T. Area (mm²/in²): 31.5/0.0489
Gage length (mm/in): 25.4/1.00

1st Cycle
Young’s Modulus: 113.8/16.49 (GPa/psi)
Δε (%): 1.604
Max σ (MPa/ksi): 238.2/34.55
Min σ (MPa/ksi): -249.6/-36.2

Mid life cycle (512)
Young’s Modulus: 89.39/12.96 (GPa/psi)
Max σ (MPa/ksi): 294.4/42.67
Min σ (MPa/ksi): -276.6/-40.09
Δε (%): 1.60
Δεf (%): 0.500
Δεp (%): 1.10

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Low Cycle Fatigue Test Data

Funding Agency: AFOSR
Specimen drawing: per ASTM E606
Machining Source: Cincinnati Testing Labs
Test temp: (513) 851-3313
Control Mode: Axial strain
Operator: Dana Swalla

Waveform: Ramp
Strain rate: 0.005 sec\(^{-1}\)
Strain range: 2.75%
Equipment: 20 kip MTS 923.14
Test Date: 1/23/02

Specimen I.D.: AR-L221

\[ N_f = 53 \] (50% load drop)
Failure location: Gage, inside extensometer

R.T. O.D (mm/in): 6.29/0.2477
R.T. Area (mm\(^2\)/in\(^2\)): 31.09/0.0482
Gage length (mm/in): 25.4/1.00

1\(^{st}\) Cycle
Young’s Modulus: 107.6/15.60
\( \Delta \varepsilon \) (%): 2.72
Max \( \sigma \) (MPa/ksi): 251.15/36.42
Min \( \sigma \) (MPa/ksi): -283.31/-41.09

Mid life cycle (27)
Young’s Modulus: assume 80.0
\( \Delta \varepsilon \) (%): 2.69 (data recorded at load peak/valley)
Max \( \sigma \) (MPa/ksi): 254.0/36.84
Min \( \sigma \) (MPa/ksi): -238.3/-34.57
\( \Delta \varepsilon \) (%): 0.457
\( \Delta \sigma \) (%): 2.29

224
Low Cycle Fatigue Test Data

Funding Agency: AFOSR
Specimen drawing: per ASTM E606
Machining Source: Low Stress Grind
Test temp: Room (± 70°F)
Control Mode: Axial strain
Operator: Dana Swalla

Waveform: Ramp
Strain rate: 0.005 sec⁻¹
Strain range: 1.0%
Equipment: 20 kip MTS 923.14
Test Date: 7/2/02

Specimen I.D.: LT5_3
Failure location: Gage, inside extensometer

Nf = 9659 (50% load drop)
R.T. O.D (mm/in): 6.35/0.25
R.T. Area (mm²/in²): 31.70/0.0491
Gage length (mm/in): 31.75/1.25

1st Cycle
Young’s Modulus: 125.8/18.2
(ＧPa/ksi): 0.97
Δe (%): 612.2/88.71
Min σ (MPa/ksi): -635.9/-92.17

Mid life cycle (4830)
Max σ (MPa/ksi): 774.8/112.3
Min σ (MPa/ksi): -464.12/-67.27
Δe (%): 1.0
Δσe (%): 0.984
Δσp (%): 0.015
**Low Cycle Fatigue Test Data**

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### 1st Cycle

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Low Cycle Fatigue Test Data

Funding Agency: AFOSR  Waveform: Ramp
Specimen drawing: per ASTM E606  Strain rate: 0.005 sec⁻¹
Machining Source: Low Stress Grind  Strain range: 2.75%
Test temp: Room (± 70°F)
Control mode: Axial strain  Equipment: 20 kip MTS 923.14
Operator: Dana Swalla  Test Date: 

Specimen I.D.: LTS-5

Nf = 384 (50% load drop)
Failure location: Gage, inside extensometer

R.T. O.D (mm/in): 6.38/0.251
R.T. Area (mm²/in²): 31.94/0.0495
Gage length (mm/in): 31.75/1.25

1st Cycle
Young’s Modulus: 117.3/16.96 (GPa/ksi)
Δε (%): 2.76
Max σ (MPa/ksi): 870.8/125.9
Min σ (MPa/ksi): -938.1/-135.6

Mid life cycle (192)
Max σ (MPa/ksi): 754.6/109.1
Min σ (MPa/ksi): -781.6/-113.2
Δε (%): 2.75
Δε₀ (%): 1.31
Δε₅ (%): 1.43

228
APPENDIX B

FRETTING TEST RESULTS
Figure B.1: SEM micrograph of fretting scar (Test 3: $\delta_s = 60 \, \mu m$, $N=10^6$ cycles).

Figure B.2: Scar topography measurement (Test 3: $\delta_s = 60 \, \mu m$, $N=10^4$ cycles).
Figure B.3: SEM micrograph of fretting scar (Test 4: $\delta_c = 120 \mu m$, $N=10^4$ cycles).

Figure B.4: SEM micrograph of fretting scar (Test 5: $\delta_c = 30 \mu m$, $N=10^4$ cycles).
Figure B.5: Scar topography measurement (Test 6: $\delta_s = 15 \mu m$, $N=10^4$ cycles).

Figure B.6: SEM micrograph of fretting scar (Test 6: $\delta_s = 15 \mu m$, $N=10^4$ cycles).
Figure B.7: Frictional force versus clip gage displacement for Test 4 ($d_0 = 120 \, \mu m$, $N = 10^4$).
Figure B.8: Frictional force versus clip gage displacement for Test 3 ($\delta_{s} = 60 \, \mu m$, $N = 10^6$).
Figure B.9: Frictional force versus clip gage displacement for Test 5 ($\Delta h = 30 \mu m, N = 10^4$).
Figure B.10: Frictional force versus clip gage displacement for Test 6 ($\delta_m = 15 \, \mu m$, $N = 10^4$).
Figure B.11: Frictional force versus clip gage displacement for Ti-5Al-2.5Sn: Test 12 ($\delta_0 = 120 \mu m$, $N = 10^6$).
Figure B.12: Frictional force versus clip gage displacement for Ti-5Al-2.5Sn: Test 14 ($\delta_t = 60$ $\mu$m, $N = 10^5$).
Figure B.13: Frictional force versus clip gage displacement for Ti-5Al-2.5Sn: Test 15 ($d = 30 \mu m$, $N = 10^5$).
Figure B.14: Frictional force versus clip gage displacement for Ti-5Al-2.5Sn: Test 16 ($\Delta z = 15 \text{ m}, N = 10^3$).
Figure B.15: Frictional force versus clip gage displacement for CP-Ti Test 23 (δₘ = 120 μm, N = 10⁵).
Figure B.16: Frictional force versus clip gage displacement for CP-Ti: Test 22($\delta_s = 60 \, \mu m, \, N = 10^6$).
Figure B.17: Frictional force versus clip gage displacement for CP-Ti Test 24 ($\delta_u = 30 \mu m$, $N = 10^4$).
Figure B.18: Frictional force versus clip gage displacement for CP-Ti: Test 25($\delta_s = 15\ \mu m$, $N = 10^4$).
APPENDIX C

EBSD RESULTS
Figure C.1: Orientation map legend (HKL Technology, 2000).

Ti-6Al-4V

Figure C.2: Orientation map and pattern quality map for Ti-6Al-4V Test 1 ($\delta_a = 60$ $\mu$m, $N = 10^5$, filename s612_2_1_nobeta).

247
Figure C.3: Orientation map and pattern quality map for Ti-6Al-4V Test 1 ($\delta_a = 60 \mu m$, $N = 10^6$), filename s612_near_test1 nobeta.
Figure C.4: Orientation map and pattern quality map for Ti-6Al-4V Test 2 ($\delta_b = 60$ $\mu$m, $N = 10^9$), filename $s612\_test2(b)\_nobody$. 
Figure C.5: Orientation map and pattern quality map for Ti-6Al-4V Test 3 ($\delta_0 = 60\ \mu m, N = 10^6$), filename s612_test3_3_nobeta.
Figure C.6: Orientation map and pattern quality map for Ti-6Al-4V Test 4 ($\delta_t = 120 \, \mu m, N = 10^4$), filename s612_test4_6_nobeta.
Figure C.7: Orientation map and pattern quality map for Ti-6Al-4V Test 5 ($\delta_x = 30 \mu m$, $N = 10^5$), filename s612_test5(h3)_nobeta.

Figure C.8: Orientation map and pattern quality map for Ti-6Al-4V Test 6 ($\delta_x = 15 \mu m$, $N = 10^5$), filename s612_test6(i)_nobeta.
Figure C.9: Orientation map and pattern quality map for Ti-6Al-4V Test 7 ($\delta_s = 60 \mu m$, $N = 10^3$), filename s612_test7_nobeta.

Figure C.10: Orientation map and pattern quality map for Ti-6Al-4V transverse cross section of as-received Ti-6Al-4V specimen, filename X_sec_t641_nobeta.
Figure C.11: Orientation map and pattern quality map for Ti-6Al-4V longitudinal cross section of as-received Ti-6Al-4V specimen. filename X_sec2_1641.

Ti-5Al-2.5Sn

Figure C.12: Orientation map and pattern quality map for Ti-5Al-2.5Sn Test 12 (δ_a = 120 μm, N = 10^6). filename ti_5_25_test12_2.
Figure C.13: Orientation map and pattern quality map for Ti-5Al-2.5Sn Test 14 ($\delta_u = 60 \, \mu m$, $N = 10^3$), filename ti_5_25_test14_3.

Figure C.14: Orientation map and pattern quality map for Ti-5Al-2.5Sn Test 15 ($\delta_u = 30 \, \mu m$, $N = 10^3$), filename ti_5_25_test15_1.
Figure C.15: Orientation map and pattern quality map for Ti-5Al-2.5Sn Test 16 ($\delta_v = 15 \mu m, N = 10^5$), filename ti_5_25_test16.

Figure C.16: Orientation map and pattern quality map for Ti-5Al-2.5Sn Test 18 ($\delta_v = 60 \mu m, N = 10^5$), filename ti_5_25_test18_1.
Figure C.17: Orientation map and pattern quality map for longitudinal cross section of as-received Ti-5Al-2.5Sn specimen, filename: ti_5_25_asrec1.
Figure C.18: Orientation map and pattern quality map for CP-Ti Test 22 ($d_0 = 60 \mu$m, $N = 10^7$), filename: CP_Ti_test22_100x.

Figure C.19: Orientation map and pattern quality map for CP_Ti Test 24 ($d_0 = 30 \mu$m, $N = 10^7$), filename: CP_Ti_test24_100x.
Figure C.20: Orientation map and pattern quality map for CP_Ti Test 25 ($\delta_n = 60$ $\mu$m, $N = 10^3$), filename: CP_Ti_test25_3_100x
APPENDIX D
EDX RESULTS
Spectrum Label: Sum Spectrum

Livetime 68.1 s

Acquisition geometry (degrees):
Tilt = 0.0
Azimuth = 0.0
Elevation = 35.0

Accelerating voltage = 10.00 kV

Total spectrum counts = 45298

<table>
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<tr>
<th>Sample data : Strobe</th>
<th>Energy (eV)</th>
<th>Rem. (eV)</th>
<th>Area</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>47.96</td>
<td>132346</td>
<td></td>
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<table>
<thead>
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<th>Optimization data : Titanium K series</th>
<th>Energy (eV)</th>
<th>Rem. (eV)</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strobe :</td>
<td>50.16</td>
<td>97136</td>
<td></td>
</tr>
<tr>
<td>Optimization element</td>
<td>31.50</td>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>

Sample is polished.
Sample is uncoated
There is a mismatch between the kV used for optimization and that of the current spectrum. The element used for optimization was Titanium.

Spectrum processing:
No peaks omitted
Processing option: All elements analyzed (Normalised)
Number of iterations = 5

Standard:
O SiO2 1-Jan-1999 12:00 AM
Al Al2O3 1-Jan-1999 12:00 AM
Ti Ti 1-Jan-1999 12:00 AM
V V 1-Jan-1999 12:00 AM

<table>
<thead>
<tr>
<th>Element</th>
<th>App Conc.</th>
<th>Intensity Corr.</th>
<th>Weight%</th>
<th>Weight% Sigma</th>
<th>Atomic%</th>
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</thead>
<tbody>
<tr>
<td>O K</td>
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<td>0.7222</td>
<td>30.64</td>
<td>2.27</td>
<td>55.36</td>
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<tr>
<td>Al K</td>
<td>3954.20</td>
<td>1.1105</td>
<td>5.98</td>
<td>0.49</td>
<td>6.41</td>
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<tr>
<td>Ti K</td>
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<td>0.9672</td>
<td>62.92</td>
<td>2.32</td>
<td>37.97</td>
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<td>V K</td>
<td>247.93</td>
<td>0.8844</td>
<td>0.47</td>
<td>1.50</td>
<td>0.27</td>
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Totals: 100.00
Spectrum Label: Sum Spectrum

Livetime 85.0 s
Acquisition geometry (degrees):
Tilt = 0.0
Azimuth = 0.0
Elevation = 35.0

Accelerating voltage = 10.00 kV

Total spectrum counts = 81692

Sample data:
Strobe:
Energy (eV)  Assm. (eV)  Area
0           58.07      334932

Optimization data: Titanium K series
Strobe:
Energy (eV)  Assm. (eV)  Area
-7.5         50.16      97136

Optimization element:

Sample is unpolished X-ray corrections may be approximate.
Sample is uncounted.
There is a mismatch between the kV used for optimization and that of the current spectrum. The element used for optimization was Titanium.

Spectrum processing:
Peak possibly skewed: 0.849 keV
Processing option: All elements analyzed (Normalised)
Number of iterations = 3

Standard:
O  SiO2  1-Jan-1999 12:00 AM
Al  Al2O3  1-Jan-1999 12:00 AM
Ti  Ti  1-Jan-1999 12:00 AM
V  V  1-Jan-1999 12:00 AM

<table>
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<th>Element</th>
<th>App Conc.</th>
<th>Intensity</th>
<th>Weight%</th>
<th>Weight% Sigma</th>
<th>Atomic%</th>
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<td>O</td>
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<td>34.68</td>
<td>2.45</td>
<td>63.74</td>
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<td>Al</td>
<td>1.0980</td>
<td>5.64</td>
<td>0.49</td>
<td>5.55</td>
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<tr>
<td>Ti</td>
<td>0.8905</td>
<td>58.48</td>
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<td>V</td>
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<td>1.48</td>
<td>-1.47</td>
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Totals: 100.00

262
Spectrum Label: Sum Spectrum

Livetime: 117.5 s

Acquisition geometry (degrees):
Tilt = 0.0
Azimuth = 0.0
Elevation = 35.0

Accelerating voltage = 10.10 kV

Total spectrum counts = 99280

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Optimization data: Titanium K series

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<th>Rem. (eV)</th>
<th>Area</th>
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</thead>
<tbody>
<tr>
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<td>-7.5</td>
<td>50.16</td>
<td>9716</td>
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</table>

Optimization element

<table>
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<th>Rem. (eV)</th>
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<tbody>
<tr>
<td>4346.0</td>
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Sample is unpolished X-ray corrections may be approximate.
Sample is uncoated
There is a mismatch between the kV used for optimization and that of the current spectrum. The element used for optimization was Titanium.

Spectrum processing:
No peaks omitted

Processing option: All elements analyzed (Normalised)
Number of iterations = 5

Standard:
O  SiO2  1-Jun-1999 12:00 AM
Al  Al2O3  1-Jun-1999 12:00 AM
Ti  Ti  1-Jun-1999 12:00 AM
V  V  1-Jun-1999 12:00 AM

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<th>Element</th>
<th>App Conc.</th>
<th>Intensity</th>
<th>Weight%</th>
<th>Weight%</th>
<th>Atomic%</th>
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<tr>
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<td>0.7338</td>
<td>32.32</td>
<td>3.59</td>
<td>57.01</td>
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<tr>
<td>Al</td>
<td>1345.37</td>
<td>1.1099</td>
<td>6.60</td>
<td>0.72</td>
<td>6.91</td>
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<tr>
<td>Ti</td>
<td>10579.93</td>
<td>0.9043</td>
<td>63.74</td>
<td>3.66</td>
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<tr>
<td>V</td>
<td>-430.91</td>
<td>0.8816</td>
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<td>2.13</td>
<td>-1.48</td>
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Total: 100.00

263
Sample: Test 12
Type: Default
ID: Ti-1.2-1.5 12

Label: Sum Spectrum
Collected: 5-Jun-2003 02:35 PM
Livetime (s): 83.20
Real time (s): 0.00
Detector: Silicon
Window: SATW
Tilt (deg): 0.0
Elevation (deg): 35.0
Azimuth (deg): 0.0
Magnification: 7122 X
Accelerating voltage (kV): 10.00
Process time: 5

Sample is unpolished X-ray corrections may be approximate.
Sample is uncorrected
There is a mismatch between the kV used for optimization
and that of the current spectrum. The element used for optimization was Titanium

Spectrum processing:
No peaks omitted

Processing option: All elements analyzed (Normalized)
Number of iterations = 5

Standard:
0 Sh 1-Jun-1999 12:00 AM
Al ADiO 1-Jun-1999 12:00 AM
Ti Ti 1-Jun-1999 2:00 AM
Sn Sn 1-Jun-1999 12:00 AM

<table>
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<th>Element</th>
<th>App</th>
<th>Intensity</th>
<th>Weight%</th>
<th>Weight%</th>
<th>Atomic%</th>
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<tr>
<td>O K</td>
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<td>22.79</td>
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<td>46.22</td>
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<td>Al K</td>
<td>2325.78</td>
<td>1.1224</td>
<td>4.93</td>
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<td>0.8250</td>
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Totals 100.00

264
Project: ti_5_25_test13
Owner: Dana
Site: Site of Interest 2

Label: Sum Spectrum
Collected: 5-Jun-2003 03:05 PM
Rietveld (s): 248.06
Real time (s): 0.00
Detector: Silicon
Window: SATW

Tilt (deg): 0.0
Elevation (deg): 35.0
Azimuth (deg): 0.0

Magnification: 3561 X
Accelerating voltage (kV): 10.00
Process time: 5

Sample is unpolished X-ray corrections may be approximate.
Sample is uncoated
There is a mismatch between the kV used for optimization and that of the current spectrum. The element used for optimization was Titanium

Spectrum processing:
No peaks omitted

Processing option: All elements analyzed (Normalised)
Number of iterations = 5

Standard:
O SiO2 1-Jun-1999 12:00 AM
Al A12O3 1-Jun-1999 12:00 AM
Sn Sn 1-Jun-1999 12:00 AM

<table>
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<th>Element</th>
<th>Conc.</th>
<th>Intensity</th>
<th>Weight%</th>
<th>Weight%</th>
<th>Atomic%</th>
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<td>0.7055</td>
<td>28.05</td>
<td>1.15</td>
<td>53.00</td>
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<td>Al</td>
<td>2186.01</td>
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<td>5.10</td>
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<td>Ti</td>
<td>22727.64</td>
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<td>0.66</td>
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Totals 100.00

265
Sample is unpolished X-ray corrections may be approximate.
Sample is uncoated
There is a mismatch between the kV used for optimization
and that of the current spectrum. The element used for optimization was Titanium.

Spectrum processing:
Peak possibly omitted: 0.940 keV
Processing option: All elements analyzed (Normalised)
Number of iterations = 5

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<th>Weight%</th>
<th>Weight% Sigma</th>
<th>Atomic%</th>
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<td>45.88</td>
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<td>0.36</td>
<td>4.62</td>
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<td>0.8255</td>
<td>0.89</td>
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Totals  100.00
Project: Project 1  
Owner: Dana  
Site: Site of Interest 1

Sample: Sample 1  
Type: Default  
ID: 

Spectrum Label: Sum Spectrum  
Live time 210.2 s  
Acquisition geometry (degrees):  
TIB = 0.0  
Azimuth = 0.0  
Elevation = 35.0  
Accelerating voltage = 10.00 kV  
Total spectrum counts = 181486

Sample data:  
Strobe: 
Optimization data: Titanium K series  
Strobe: 
Optimization element:

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<th>Energy (eV)</th>
<th>Resm. (eV)</th>
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</tr>
<tr>
<td>-7.5</td>
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<tr>
<td>-4540.0</td>
<td>31.50</td>
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Sample is unpolished X-ray corrections may be approximate.
Sample is uncoated
There is a mismatch between the kV used for optimization and that of the current spectrum.
The element used for optimization was TitaniumK.

Spectrum processing:  
No peaks omitted  
Processing option: All elements analyzed (Normalised)  
Number of iterations = 4

Standard:  
O SiO2 1-Jun-1999 12:00 AM  
Al AD03 1-Jun-1999 12:00 AM  
Ti Ti 1-Jun-1999 12:00 AM  
V V 1-Jun-1999 12:00 AM

<table>
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<th>Element</th>
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<th>Weight%</th>
<th>Weight% Sigma</th>
<th>Atomic%</th>
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<td>0.25</td>
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Total: 100.00

267
Specimen Label: Sam Spectrum

Livetime: 210.2 s

Acquisition geometry (degrees):
Tilt = 0.0
Azimuth = 0.0
Elevation = 55.0

Accelerating voltage = 10.00 kV

Total spectrum counts = 182015

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<th>Rem. (eV)</th>
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<tbody>
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Optimization data: Titanium K series

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<th>Rem. (eV)</th>
<th>Area</th>
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<tbody>
<tr>
<td>Strobe</td>
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<td>30.16</td>
<td>97136</td>
</tr>
<tr>
<td>Optimization element</td>
<td>4340.0</td>
<td>31.50</td>
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</tr>
</tbody>
</table>

Sample is unpolished and corrections may be approximate.
Sample is uncoated.
There is a mismatch between the kV used for optimization and that of the current spectrum. The element used for optimization was Titanium.

Specimen processing:
No peaks omitted.

Processing option: All elements analyzed (Normalised)
Number of iterations = 4

Standard:
O SiC2 1-Jun-1999 12:00 AM
Al Al2O3 1-Jun-1999 12:00 AM
V V 1-Jun-1999 12:00 AM

<table>
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VITA

Dana Ray Swalla was born in Lancaster, California on September 17, 1964. She graduated from Palm Bay High School in Melbourne, FL in 1982. After spending a few years traveling the country, Dana returned to her adopted home state to attend college. Most of her coursework was completed part-time while working full-time as a mechanical designer, which enabled her to graduate debt-free from the University of Florida with High Honors in 1993. She began graduate work at the Georgia Institute of Technology in Atlanta, Georgia in September, 1997, after working in industry as a mechanical engineer for a number of years. Dana was awarded the degree Master of Science in June 1999 and officially began her PhD program in January, 2001.