### ABSTRACT

Title of Dissertation:EFFECT OF MICROSTRUCTURE ON THE ROOM<br/>TEMPERATURE TENSILE AND CREEP<br/>DEFORMATION MECHANISMS OF α-β TITANIUM<br/>ALLOYSAllan Wayne Jaworski Jr., Doctor of Philosophy, 2005

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Two-phase  $\alpha$ - $\beta$  titanium alloys are used in many applications because of their high specific strength, corrosion resistance, processability, and biocompatibility. The room temperature tensile and creep deformation mechanisms of  $\alpha$ - $\beta$  alloys must be understood in order to design alloys with desired properties and improved creep resistance. There is a lack of understanding in this regard. The aim of this investigation is to systematically study the effects of microstructure, stability of the  $\beta$  phase, and alloying elements on the deformation mechanisms of  $\alpha$ - $\beta$  titanium alloys using Ti-6.0wt%Mn and Ti-8.1wt%V as the model systems.

The tensile and creep deformation mechanisms and microstructure were studied using SEM, TEM, HREM, and optical microscopy. In addition, theoretical modeling was performed in terms of crystallographic principles and stress analysis.

It was found for the first time in an  $\alpha$ - $\beta$  titanium alloy (Ti- 8.1wt%V) that the  $\alpha$ phase deforms by twinning and the  $\beta$  phase deforms by stress induced martensite, different mechanisms than the single-phase  $\alpha$  and  $\beta$  alloys with similar grain size. Singlephase  $\alpha$  deforms predominantly by slip, and single-phase  $\beta$  deforms predominately by twinning. This is also the first time that stress induced martensite has been observed in a creep deformed  $\alpha$ - $\beta$  titanium alloy. However in the case of Ti-6.0wt%Mn, where the  $\beta$  phase stability is higher, stress induced martensite was not observed.

The deformation mechanisms are modeled in terms of the  $\beta$  phase stability and interactions between phases, including elastic interaction stresses,  $\alpha$  phase templating, interactions of deformation products, and  $\alpha$ - $\omega$  interactions. A model is also proposed which explains anisotropic interface sliding based on locking of growth ledges.

These results are extremely valuable when designing new alloys with improved resistance to creep and other failure modes. The observed deformation mechanisms can directly affect the mechanical reliability of systems. For instance, increased creep strain can alter the dimensional tolerances of components and the observed stress induced products can act as nucleation sites for fracture initiation and stress corrosion cracking.

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# EFFECT OF MICROSTRUCTURE ON THE ROOM TEMPERATURE TENSILE AND CREEP DEFORMATION MECHANISMS OF $\alpha$ - $\beta$ TITANIUM ALLOYS

by

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# **Chapter 1**

# Introduction

Titanium alloys are used in a wide variety of applications, including defense, aerospace, biomedicine, industry and consumer products. Titanium is valued for its high specific strength, corrosion resistance and biocompatibility. Its higher cost compared to aluminum or steel is justified by its performance in critical applications. In many of these applications two phase  $\alpha$ - $\beta$  titanium alloys are the material of choice due to their combination of high strength and processability. In order to optimize these properties while maximizing reliability the study of tensile and creep deformation mechanisms of  $\alpha$ - $\beta$  titanium alloys is necessary. The mechanical properties of the two-phase alloys are dependent on several factors, including the properties of their component phases, microstructure, and interactions between phases. A review of the microstructure, tensile and creep deformation mechanisms of titanium alloys is given below. The aim of this investigation follows.

#### 1.1. Background

#### 1.1.1 α and β phases

The  $\alpha$  phase of titanium has a hexagonal close packed (hcp) structure. The  $\beta$  phase has a body centered cubic (bcc) structure. Pure titanium undergoes an allotropic transformation from the  $\alpha$  to  $\beta$  phase as its temperature is increased through 1156 K (883°C). Elements that produce little change in the transformation temperature or cause it to increase are called  $\alpha$  stabilizers, and are generally simple metals or interstitial

elements. Elements that stabilize the  $\alpha$  phase include Al, Ga, Sn and O. Elements that decrease the transformation temperature are referred to as  $\beta$  stabilizers, and are generally transition metals and noble metals. Some common  $\beta$  stabilizers are V, Nb, Ta, Mo and Mn [1].

Titanium alloys can have a single phase  $\alpha$  or  $\beta$  microstructure, or a two-phase  $\alpha$ - $\beta$ microstructure of several morphologies. Single-phase  $\alpha$  titanium can be commercially pure (CP) or an alloy containing quantities of  $\alpha$  stabilizing elements listed above. Single phase  $\alpha$  and near  $\alpha$  titanium alloys are characterized by satisfactory strength, toughness, creep resistance and are weldable. Furthermore they lack a ductile to brittle transformation, and thus are suitable for cryogenic applications [1]. Single-phase  $\beta$ alloys typically contain one or more  $\beta$  stabilizing elements. They have a higher strength to weight ratio than the  $\alpha$  alloys. They are however, as are other alloys with a BCC structure, unsuitable for cryogenic applications due to a ductile to brittle transformation. Due to difficulties in processing and the inherent instability of the  $\beta$  phase, the commercial application of  $\alpha$  and  $\alpha$ + $\beta$  titanium alloys far surpasses that of  $\beta$  alloys.

Alloys that contain both the  $\alpha$  and  $\beta$  phases are called  $\alpha$ - $\beta$  alloys. Usually  $\alpha$ - $\beta$  alloys contain a mixture of  $\alpha$  and  $\beta$  stabilizers. The most common and widely studied example is Ti-6Al-4V. The two-phase  $\alpha$ - $\beta$  alloys can also be  $\beta$  stabilized, whereby sufficient  $\beta$  stabilizers are added to retain the  $\beta$  phase upon quenching, and the  $\alpha$  phase is nucleated by heat treatment in the  $\alpha$ + $\beta$  field. The model  $\alpha$ - $\beta$  alloys used in this investigation are  $\beta$  stabilized binary alloys.

Many different  $\alpha+\beta$  microstructures can be achieved by alloying and thermomechanical processing. The morphology, volume fraction and distribution of the

phases can be altered by processing. Primary  $\alpha$  is formed by nucleation and growth from the  $\beta$  phase. Heavily working Ti alloys in the  $\alpha+\beta$  field results in an equiaxed (globular)  $\alpha$ phase microstructure. When the alloy is processed above the  $\beta$  transus temperature, a Widmanstätten (acicular) microstructure consisting of elongated plates or needles of the  $\alpha$ phase can form by nucleation and growth if cooling rates are slow, or hexagonal martensite can form if the alloy is quenched [2]. In alloys that are cooled slowly the Widmanstätten plates can form in colonies of plates which all have the same orientation. With increased cooling rates random mixtures of plate orientations form in the same areas, which is referred to as a basketweave structure [2]. Alloys worked in the  $\beta$  field (Widmanstätten) have superior fracture toughness but lower ductility than those worked in the  $\alpha+\beta$  field (equiaxed) [3]. A duplex microstructure also exists that has a combination of equiaxed and elongated  $\alpha$  grains, and is produced by an initial heat treatment high in the  $\alpha+\beta$  field, then a subsequent treatment at a lower solution temperature [3].

#### 1.1.2 $\beta$ phase stability

The stability of the  $\beta$  phase is an important property in single-phase  $\beta$  and twophase  $\alpha$ - $\beta$  alloys. As discussed above, the temperature above which the  $\beta$  phase is stable in titanium can be altered by the addition of  $\alpha$  and  $\beta$  stabilizers. The lower this temperature is, the more stable the  $\beta$  phase will remain after quenching. In order to quantify this stability, the molybdenum equivalency (MoE) can be determined for any  $\beta$ alloy with any number and concentration of  $\alpha$  and  $\beta$  phase stabilizers [4]. Molybdenum was chosen as a reference  $\beta$  stabilizing element, and the MoE of an alloy is given as the

volume % of Mo in a Ti-Mo alloy with the same stability. Generally a MoE of about 10 is required to stabilize the  $\beta$  phase [4].

The  $\beta$  phase stability can affect the tensile and creep deformation mechanisms, thus affecting the tensile and creep behavior. In general, as stability is increased, the tensile deformation mechanisms of single-phase  $\beta$  Ti alloys will change from stress induced martensites to twinning and finally slip for alloys with the highest stabilities [5, 6].

#### **1.1.3** ω phase

The  $\omega$  phase is a metastable phase in Ti-alloys at room temperature and pressure. The  $\omega$  phase forms as a nanostructured phase due to the instability of  $\beta$  titanium alloys, and is dispersed throughout the  $\beta$  phase. The  $\omega$  phase can also be formed from the  $\alpha$  phase under high hydrostatic pressures at elevated temperatures [7-10]. In the context of the current work this transformation only becomes important when the reverse  $\omega \rightarrow \alpha$  transformation is considered, which is discussed in Chapter 7.

The  $\omega$  phase has a P6/mmm hexagonal structure (Strukturbericht C32 designation) [7,11,12]. Omega phase which forms in the  $\beta$  phase can be athermal, which occurs by a diffusionless transformation upon quenching which cannot be suppressed, or thermal, in which it grows after a period of aging at elevated temperatures [13-15]. Athermal  $\omega$  phase has been observed in the  $\beta$  phase of numerous Ti – transition metal alloys, which are reviewed by Collings [1]. Ellipsoidal and cubic shaped crystallites have been observed [16]. Four orientations of the  $\omega$  phase can exist simultaneously in the  $\beta$  phase, with orientations to the  $\beta$  phase of the type  $(0001)[2\overline{1}\overline{1}0]_{\omega}$  //{111}/ $10\rangle_{\beta}$  [17]. The

crystallography of the  $\omega$  phase and its effects on  $\beta$  phase deformation are discussed in Chapter 7.

#### 1.1.4 Low temperature deformation mechanisms of titanium alloys

The low temperature (<.25T<sub>m</sub>) plastic deformation of titanium alloys can occur by several mechanisms. Slip and twinning can occur in  $\alpha$  and  $\beta$  alloys, and stress induced martensite can occur in  $\beta$  alloys.  $\alpha$ - $\beta$  titanium alloys can deform in the  $\alpha$  or  $\beta$  phases by the above mechanisms, or by sliding between phases. Below is a short review of the low temperature deformation mechanisms of titanium alloys.

The  $\alpha$  phase can deform by slip during tensile or creep deformation. The types of possible slip include 'a' type slip with a Burgers vector  $\vec{b} = 1/3 \langle 11\bar{2}0 \rangle_{\alpha}$  on the basal  $(0001)_{\alpha}$  or prism planes  $\{10\bar{1}0\}_{\alpha}$ , 'c' type slip with a Burgers vector  $\vec{b} = \langle 0001 \rangle_{\alpha}$  on  $\{h\bar{k}i0\}_{\alpha}$  prism planes, and 'c+a' slip with  $\vec{b} = \langle 11\bar{2}3 \rangle_{\alpha}$  on  $\{10\bar{1}l\}_{\alpha}$  pyramidal planes [18, 19]. An increase in the amount of  $\alpha$  stabilizing elements can affect the deformation behavior of  $\alpha$  alloys. An increase in Al content up to 5% tends towards screw dislocations [20]. Higher concentrations tend toward localization of slip, resulting in planar slip bands [21]. Slip is most common with 'a' type dislocations on prism planes [18]. Tensile and creep deformation in the  $\alpha$  phase can also occur by twinning. The four most common twins are of the types  $\{10\bar{1}2\}\langle\overline{1}011\rangle$ ,  $\{10\bar{1}1\rangle\langle\overline{1}012\rangle$ ,  $\{11\bar{2}2\}I/3\langle11\bar{2}3\rangle$ , and  $\{11\bar{2}1\}I/3\langle\overline{1}\bar{1}26\rangle$  [19].

Plastic deformation of the  $\beta$  phase can occur by slip, twinning, or by several types of stress induced martensite. Slip in the  $\beta$  phase is most common on the  $\langle 1 \overline{1} 1 \rangle \langle 1 1 0 \rangle_{\beta}$  slip

system. Both  $\{112\}\langle 111\rangle$  [5,22] and  $\{332\}\langle 113\rangle$  [13,23-25] twins have been observed in Ti-V, Ti-Mo, Ti-Cr, and Ti-Mo-Zr alloys. In the single phase Ti-14.8V alloy studied by Ramesh, which has the same chemistry as the  $\beta$  phase of the  $\alpha$ - $\beta$  Ti-8.1V alloy studied in this investigation,  $\{332\}\langle 113\rangle$  twinning was observed following tensile deformation [25].

Microstructure affects the deformation mechanisms described above. A decrease in grain size increases the stress required for twin nucleation [26]. In fine grains the stress required for slip is less than that for twinning, therefore slip is the preferred deformation mechanism in fine grains. The primary deformation mechanism changed from twinning to slip when grain sizes were reduced in single phase  $\alpha$  and  $\beta$  Ti-Mn and Ti-V alloys, and twinning was not observed in the alloys with smallest grain sizes [27-29]

Stress induced martensite can occur in metastable  $\beta$  alloys where the  $\beta$  phase stability is sufficiently low [30]. In general, martensite nucleates at temperatures below the martensite start (M<sub>s</sub>) temperature, and the application of stress effectively raises the M<sub>s</sub> temperature [30-32]. Several types of stress induced martensite have been reported in  $\beta$  titanium alloys, hexagonal close packed ( $\alpha'$ ), orthorhombic ( $\alpha''$ ), face-centered orthorhombic, and face-centered cubic.

The  $\alpha'$  hexagonal martensite is the most common and widely reported martensite. It can form by quenching or can be stress induced. It has been observed to form during quenching in commercially pure titanium as well as Ti-5V and Ti-10V [6], Ti-7.5wt%V [33] and Ti-12wt%V [34]. Koul and Breedis identified hexagonal stress induced martensite in both Ti-13Mo and Ti-16V deformed in tension [32]. Two hexagonal martensites are described by Hammond and Kelly [35] in Ti-5Mn, one with a  $\{334\}_{\beta}$ habit plane and smooth interface and one with a  $\{344\}_{\beta}$  habit plane and a zig-zagged

interface. Hexagonal martensites with a  $\{344\}_{\beta}$  habit plane are internally  $\{10\overline{1}1\}\langle\overline{1}012\rangle$  twinned [6, 35, 36].



**Figure 1.1.** Crystal structures and slip planes of the  $\alpha$  (hcp) and  $\beta$  (bcc) phases of titanium.

Stress induced orthorhombic martensite ( $\alpha''$ ) was first reported by Bagariatskii as part of an extensive investigation of martensites in Ti-V alloys by x-ray and hardness measurements. He reported the existence of  $\alpha''$  near the Ti-10wt%V composition [37]. Orthorhombic martensite was identified during the tensile deformation of Ti-20Mo [24]. In Ti-20V deformed in tension  $\alpha''$  was identified by x-ray diffraction [6]. Grosdidier reports  $\alpha''$  in  $\beta$ -Cez (a proprietary alloy) deformed in tension, while in heavily deformed specimens of the same alloy  $\alpha''$  transforms into  $\alpha'$  [30, 38]. Additions of Al to Ti-Mo and Ti-V alloys promotes  $\alpha''$  [16] A face centered orthorhombic martensite, which has a different axial ratio than  $\alpha''$ , was reported for Ti-12.6V by Oka et. al. [39], but Williams [16] attributed this phase to thin film processing effects, and was unable to reproduce the face centered orthorhombic martensite in Ti-11.6V. Face centered cubic (fcc) martensites have been reported [40], but these have not been identified in the bulk and have been dismissed as a spontaneous transformation product due to thinning for electron microscopy [16].

In addition to the stress induced martensites, stress induced  $\omega$  phase plates have been observed during tensile deformation of Ti-15V and Ti-19V [17, 24]. These plates were one variant of the  $\omega$  phase that grew by the consumption of the other  $\omega$  variants and  $\beta$ . A small amount of hexagonal martensite,  $\alpha'$ , was found within these plates. It was suggested that the  $\omega$  phase is an intermediate phase in the stress induced transformation  $\beta \rightarrow \alpha'$  transformation.

#### **1.1.5 Plastic deformation in α-β alloys**

The deformation mechanisms of  $\alpha$ - $\beta$  alloys depend on several factors, including the properties of the component phases, their morphology and the volume fraction of phases. The orientation and interface between phases must also be considered. When the two-phase alloys are subjected to stress, the component phases deform differently, resulting in interactions between phases. These interactions can occur during elastic, elasto-plastic, or plastic deformation [41]. Therefore, deformation in the  $\alpha$ - $\beta$  alloys is a complicated process which cannot be modeled solely on the  $\alpha$  and  $\beta$  phase deformation behavior and laws of mixing. Two factors which must be considered are the orientation relationship between phases and the interface morphology. For instance, in alloys with a Burgers orientation relationship between the  $\alpha$  and  $\beta$  phases slip has been observed to cross the  $\alpha$ - $\beta$  interface due to an alignment of slip systems in the  $\alpha$  and  $\beta$  phases [42-48]. Sliding can occur between the  $\alpha$  and  $\beta$  phases during tensile and creep deformation of

two-phase titanium alloys. This is termed interphase interface sliding and has been observed to occur during room temperature (RT) tensile and creep deformation by several researchers [27, 45, 49, 50].

#### **1.1.6** Low temperature creep in titanium

Creep is usually thought of as a high temperature phenomenon. At high temperatures creep deformation is controlled primarily by diffusion in the bulk and along grain boundaries [51-53]. One characteristic of titanium alloys is that they are prone to creep at low homologous temperatures ( $<0.25T_m$ ) and at low stresses, in some cases as low as 25-40% of their 0.2% yield stress (YS) at room temperature [43, 55, 56]. This can cause failure in components by a change in dimensional tolerances or rupture if creep reaches the tertiary stage which was reported by Luster [56], although low temperature creep is generally of the transient type [42-48, 54]. The creep deformation mechanisms of titanium alloys deformed at low temperatures are different than those at high temperatures. Therefore, it is important to understand the creep deformation mechanisms of titanium alloys at low temperatures in order to design alloys for improved low temperature creep resistance.

Significant creep of commercially pure (CP) titanium at room temperature (RT) was first reported in 1949 [55]. Creep in CP Ti and  $\alpha$  alloys has primarily been attributed to slip [46]. Twinning has also been observed during creep of  $\alpha$  titanium alloys when the grain size was large, and can be instantaneous of time dependent. Time dependent twinning was first observed by Ankem et. al. in  $\alpha$  titanium alloys [57, 58].

In comparison to studies of low temperature creep of CP and  $\alpha$  alloys, studies of the room temperature creep of  $\beta$  and near  $\beta$  titanium alloys has been quite limited, with the exception of studies on  $\beta$  Ti-Mn and Ti-V alloys performed by Ankem et. al. [27, 59-61]. The creep strain of  $\beta$  alloys was found to depend on their stability and grain size. The creep strain was greater in alloys with lower stability and larger grain size due to additional strain from twinning, which did not occur in the higher stability alloys with small grains. Time dependent twinning was also observed [60].

Creep studies in  $\alpha$ - $\beta$  titanium alloys have primarily concentrated on several commercial alloys, Ti-6Al-4V [44, 49], Ti-5Al-2.5Sn(-0.5Fe) [43, 47, 48], Ti-6Al-2Nb-1Ta-0.8Mo [45], Ti-6Al-2Cb-1Ta-0.8Mo [42] and Ti-6Al-2Sn-4Zr-2Mo [46]. Creep in  $\alpha$ - $\beta$  alloys has been overwhelmingly attributed to slip in the  $\alpha$  and  $\beta$  phases, as well as slip across the  $\alpha$ - $\beta$  boundaries due to the alignment of slip systems. The amount of creep strain is related to the colony size, which affects slip length [45]. A model has been developed for the mechanism of slip transmission by Suri et. al. [48]. Stress induced martensite has not been reported as a creep deformation mechanism in  $\alpha$ - $\beta$  titanium alloys.

#### **1.2.** Aim

The scope of the work contained within this dissertation expanded and took new directions as novel aspects of the tensile and creep deformation behavior of the model  $\alpha$ - $\beta$  Ti-Mn and Ti-V alloys became apparent. The initial and primary goal was an investigation of the tensile and creep deformation mechanisms of  $\alpha$ - $\beta$  Ti-Mn and Ti-V alloys with a Widmanstätten microstructure. After extensive TEM examinations of these

alloys, it became apparent that the deformation mechanisms of the  $\alpha$ - $\beta$  alloys were vastly different than expected based on previous studies of the single phase alloys with the same chemistry as the component  $\alpha$  and  $\beta$  phases of the two-phase alloys. These differences included twinning in fine  $\alpha$  grains and stress induced hexagonal martensite formation in the  $\beta$  phase of tensile and creep tested specimens. The reasons for this difference in deformation mechanisms between the single and two-phase alloys were explored and a model based on interactions between the  $\alpha$  and  $\beta$  phases and  $\beta$  phase stability was developed to explain these newly observed two-phase deformation mechanisms. The interactions included the effect of elastic interactions stresses, the  $\alpha$  phase acting as a template for the nucleation of hexagonal martensite in the  $\beta$  phase, and the resolution of shear stresses from  $\alpha$  and  $\beta$  phase deformation products onto the adjacent phase.

The  $\alpha/\beta$  interface structures of the model two-phase Ti-Mn and Ti-V alloys with Widmanstätten structure were investigated in an attempt to explain and model the interphase interface sliding behavior of the alloys during tensile and creep deformation. Of particular interest is the anisotropic interface sliding of Ti-Mn alloys, which had not been explained satisfactorily.

In order to further examine the effect of  $\alpha$  phase microstructure on the  $\alpha$  and  $\beta$  phase deformation mechanisms, the tensile deformation mechanisms of Ti-8.1V with an equiaxed  $\alpha$  phase microstructure were studied. It was unknown if the interactions proposed for the Widmanstätten microstructure would occur in the alloy with equiaxed microstructure. If similar interactions occurred, then the deformation mechanisms would resemble those of the  $\alpha$ - $\beta$  alloy with Widmanstätten microstructure, otherwise the deformation mechanisms were expected to be similar to those of the single-phase alloys.

Finally, the contribution of the nanoscale  $\omega$  phase to the tensile and creep deformation behavior of  $\alpha$ - $\beta$  alloys was explored. The  $\omega$  phase is present in the  $\beta$  phase of Ti-8.1V with Widmanstätten and equiaxed microstructures. It was hypothesized, when present in the  $\beta$  phase of an  $\alpha$ - $\beta$  titanium alloy, the  $\omega$  phase affects the  $\beta$  phase deformation mechanisms through  $\alpha$ - $\omega$  interactions. The results of this part of the investigation further explain the deformation mechanisms observed in Ti-8.1V with both Widmanstätten and equiaxed microstructures.

# Chapter 2

# **General Procedures**

The following are general procedures used in the course of this investigation. They are referenced as required in Chapters 3-7 for individual investigations.

#### 2.1. Heat treatment

The Ti-6.0wt%Mn and Ti-8.1wt%V alloys used in this investigation were melted as 13.6 kg ingots and processed to 1.74 cm diameter bars at the RMI company in Niles, Ohio. Rolling to a 60% reduction in area was carried out in the  $\alpha$ + $\beta$  field at 973 K.

In order to achieve the desired  $\alpha$ - $\beta$  Widmanstätten and equiaxed microstructures, the alloys were heat treated in two ways. Prior to heat treatment the alloys were sealed in quartz tubes evacuated to  $10^{-3} - 10^{-4}$  Pa in order to prevent oxidation. To achieve the Widmanstätten microstructure of Ti-6.0Mn and Ti-8.1V, the alloys were heat treated for 2 hours at 1173 K (above the  $\beta$  transus), then furnace cooled to 963 K and annealed for 200 hours at this temperature, followed by a water quench [27]. To achieve an equiaxed  $\alpha$ phase microstructure for Ti-8.1V the alloy was heat treated at 1018 K (just below the  $\beta$ transus) for 168 hours, then furnace cooled by 10K every 48 hours to 963 K. The alloy was aged at this temperature for 200 hours, followed by a water quench. To maintain an equiaxed microstructure it was important to keep the temperature below the  $\beta$  transus temperature of 1033 K [62], which necessitates a long treatment time to grow the  $\alpha$ grains. If the alloy was heated above this temperature, the  $\alpha$  phase would go into solution,

and the result would be a Widmanstätten, not equiaxed,  $\alpha$  phase microstructure upon cooling below the  $\beta$  transus.

Final heat treatment of the two-phase alloys examined in this study as well as the single phase alloys examined in previous studies was at the same temperature of 963 K for 200 hours. This time is sufficient for thermodynamic equilibrium to be reached. The result is that the solute concentrations of the  $\alpha$  and  $\beta$  phases of the two-phase alloys are equivalent to the solute concentrations of the single-phase  $\alpha$  and  $\beta$  alloys, which lie on the same tie line. This relationship as well as the chemistry and compositions of the single and two-phase alloys are illustrated by the Ti-V and Ti-Mn phase diagrams [62, 63] in Appendix A.

#### 2.2. Tensile and creep specimen preparation

Creep and tensile specimens were machined to the specifications given in Appendix B. Electric discharge machining was used to cut the flats along the gage length to minimize any possible deformation that may be induced by conventional milling. The flats were then polished by hand with 600-1200 grit silicon carbide paper mounted on a flat aluminum bar in order to maintain a flat specimen surface.

Electropolishing was used as a final finish on specimens with a Widmanstätten microstructure [27]. Specimens were polished at 213 K in a solution of 92.5% methanol, 5% H<sub>2</sub>SO<sub>4</sub>, and 2.5% HF. This process comes with the risk of heavily pitted specimens, and a low success rate. Therefore, Buehler Mastermet<sup>®</sup> colloidal silica polishing suspension on a Chemomet<sup>®</sup> pad was used for the final polishing of the tensile specimen with equiaxed microstructure. The Beuhler colloidal silica polish was used for its 100%

reliability and ultimately superior, mirror-like final polish. The reliability was particularly important, as only a very limited quantity of Ti-8.1V was available for the equiaxed study. It is recommended that students working with titanium alloys in the future use this polish in lieu of electropolishing. The specimens were then etched with R etch and A etch (see Appendix C) to reveal grain structure.

#### 2.3. Attachment of fiducial grids

Gold-palladium fiducial lines were deposited on the polished specimen surfaces. The fiducial grid serves several purposes: to allow easy identification of the same area for pre and post testing microscopy, to measure strain in local areas, and to aid in the identification of interphase interface sliding, indicated by displacement of lines across boundaries [64].

Details of the procedure used to draw gold lines on the Ti-6.0Mn and Ti-8.1V specimens with a Widmanstätten microstructure are given in [27]. Lines were drawn with a 20  $\mu$ m spacing. A similar procedure was used for the specimen with equiaxed microstructure. Shipley Microposit 2400 PMMA positive electron resist was spun onto the specimen surface at 3500 RPM for 50 seconds in a UV free clean room and pre-baked at 170°C for 30 minutes. The specimens were transported in aluminum foil and a dark box to guard the resist from light. A JEOL 840 microprobe was used to expose the resist. The grid line spacing was increased to 50  $\mu$ m for this alloy to allow for the slightly larger  $\alpha$  grain size and to allow better viewing of the  $\alpha$  and  $\beta$  deformation mechanisms between grid lines, which was the primary focus. Beam writing conditions were 25kV, 25x magnification and an aperture size of 4. Test lines were drawn with beam currents

ranging from 2-100nA. Lines were drawn on the specimen with beam currents of 10 and 100 nA. The most reliable beam current to insure adequate exposure, albeit with slightly thicker lines, is 100nA. The resist was then developed for one minute. The specimen surface was etched lightly for 15 seconds by dilute titanium etch (Appendix C) to facilitate removal of the resist. Gold-palladium was deposited to a thickness of 20 Å by an Anatech LTD Hummer X sputtering system. Acetone, rather than the Microchem Nanoremover PG used by Greene [27], was used to remove the remaining photoresist after the deposition of gold-palladium grid lines under sonication for 10 minutes with great success.

#### 2.4. Tensile testing

Tensile testing was conducted at room temperature in an Instron 6200 floor model servo-hydraulic materials testing machine with a 250KN load cell. Testing was performed at a strain rate of  $3.28 \times 10^{-5}$ /sec to a total strain of 3% for all specimens. An Instron clip on extensometer with a 12.7 mm gauge length and 5% maximum extension was used to measure strain.

#### 2.5. Creep testing

Specimens examined for this work were creep tested in an ATS lever arm creep test machine at 95% of the measured yield stress over a period of 278 hours [27]. A clip on extensometer was used to continuously collect strain data over the test period.

#### 2.6. SEM and optical microscopy

SEM micrographs were taken of the polished specimen surfaces prior to and following tensile and creep deformation. This procedure is useful for separating deformation products that arise during tensile and creep testing from those that may be present prior to testing. A JEOL 840 SEM was used to record images of the alloys with Widmanstätten microstructure [27], and an Electroscan ESEM was utilized for observations of the Ti-8.1V tensile specimen with equiaxed microstructure. A Zeiss ICM 405 inverted metallographic light microscope was used to photograph the surface of each specimen following deformation.

#### 2.7. TEM sample preparation

The success of transmission electron microscopy (TEM) depends on the careful preparation of specimens, which must (1) remain unadulterated and (2) be sufficiently thin for electron transparency. There are many common preparation methods, some better suited to the two-phase metallic specimens than others for a variety of reasons. Several basic steps are involved for the preparation of specimens from the  $\alpha$ - $\beta$  titanium tensile and creep samples. These are outlined below.

#### 2.7.1 Cutting TEM samples from creep or tensile specimens

The first step is removal of a specimen from the bulk material. A Buehler Isomet Low Speed Saw with diamond wafering blade was used to slice sections of material from the bulk, from both the deformed material in the gage length and the undeformed material in the threaded ends of the tensile and creep specimens. Buehler Isocut<sup>®</sup> fluid was used to
lubricate and cool the wafering blade while removing debris, and a saw speed of 5 was selected. Cutting speeds were kept slow to prevent sample damage due to cutting forces. One slice was made initially to reveal the interior of the sample material of interest, then subsequent slices were made by moving the sample holder a set distance in relation to the blade. This is accomplished by dialing in the attached micrometer the desired displacement. 18 divisions of the micrometer yielded specimens of approximately 120 microns thick.

Care must be taken when cutting slices from the undeformed, threaded ends of the tensile or creep specimen. The threaded surface, when lowered against the wafering blade, can cause the blade to deflect to one side of the thread, making it impossible to cut thin slices of even thickness, and possibly damaging the blade. For this reason the threads should be filed flat on the contact surface prior to lowering the specimen to the wafering blade.

A Gatan model 659 disc punch was then used to produce 3 mm discs from the cut slices. Four discs could be produced from each undeformed (threaded end) slice, and one disk from each deformed (reduced section) slice. The Gatan punch is designed such that no additional deformation is introduced into the specimen.

#### 2.7.2 Pre-thinning

The 3mm disk must be pre-thinned using mechanical polishing techniques. Two separate techniques were used to achieve the best possible specimens. Dimpling and tripod polishing were used in combination to produce a greatly reduced thickness of material. Prior to dimpling , tripod polishing was used to achieve a flat specimen with

uniform specimen thicknesses for all specimens. This process also served to remove scratch marks on the surface of the samples resulting from the wafering blade. Diamond lapping films as fine as 1 µm were used for this operation initially, but it was found that 800 grit silicon carbide paper was sufficiently fine for initial thinning. One side of the sample is first mounted using mounting wax, and polished flat to remove scratch marks, then flipped and remounted on its opposite side onto the tripod polisher. A procedure known as high angle wedge polishing [65] was then used to monitor the progress of the sample thinning without removing the sample from the tripod. In this procedure the lengths of the legs of a modified tripod (the Quadripod) are calibrated such that a 15, 30, or 45° angle is polished onto the edge of the sample. These legs are then removed and the sample polished flat to reduce the thickness. Using a microscope and trigonometry, the horizontal length of the angled facet is measured and correlates to the specimen thickness. This apparatus and the TEM specimen profile are shown below in Figure 2.1 [65]. Tripod polishing with depth monitoring was typically carried out to produce specimens ~80 µm thick. This technique can be used to accurately thin specimens to a thickness of  $<10 \,\mu\text{m}$  without subsequent preparation before final thinning, but it was found that the titanium specimens curled or bent when this thin. Therefore, dimpling was used to produce a self-supporting specimen with a thicker edge.



**Figure 2.1.** Schematic of "Quadripod" apparatus for tripod polishing with depth monitoring. At right is the specimen geometry, where 'x' is measured under a microscope to calculate specimen thickness 'y' [65].

Dimpling was performed on South Bay Technologies SBT515 dimpling machine. The dimple depth is difficult to ascertain exactly, but through a combination of using the attached depth gauge and measurement of the dimple width and correlating with the dimple depth (method and chart in Appendix D), an estimate of the dimple depth could be made. Usually estimates of the dimple depth by measurement of the dimple width overestimated the dimple depth due to imperfect specimen centering on the base. Typically dimple depths of ~30  $\mu$ m, resulting in a thinned area <20  $\mu$ m thick, could be obtained when dimpling to the edge of both sides of a well centered 3mm sample.

## 2.7.3 Final thinning

Final thinning can be performed in two ways, jet polishing or ion milling. Jet polishing is a chemical process that uses a combination of an acid containing electrolyte and electric current to remove material from the sample. It is relatively rapid, but etches

the  $\alpha$  phase of the two-phase titanium alloys more rapidly than the  $\beta$  phase. The result is a thickness difference at the  $\alpha$ - $\beta$  interface that makes any study of the interface, as well as identification of any features crossing it, impossible. Additionally, jet polishing can introduce a hydride phase at the  $\alpha$ - $\beta$  interface of titanium alloys [66, 67]. For these reasons ion milling should be used for two-phase titanium alloys. The exception to this would be when heating of the sample is a concern, for example when examining a metastable  $\beta$  phase, where high temperatures may induce the formation of thermal  $\omega$  phase into an alloy that previously did not have any. This would give misleading information on the presence of  $\omega$  phase, and therefore a specimen prepared by jet polishing should be examined in order to assess the presence of  $\omega$  phase within the  $\beta$  phase.

A few Ti-6.0Mn specimens were prepared by jet polishing using a Fischione Instruments Model 120 twin-jet electropolisher to examine the  $\beta$  phase for the extent of  $\omega$ phase formation. An electrolyte solution of 92.5% methanol, 5% sulfuric acid and 2.5% hydrofluoric acid cooled to 203 K in a dry ice / methanol bath, a pump speed of 6 and a current of 30 mA were used.

In the course of this study, ion milling with a Baltech Rapid Etching System RES010 was used overwhelmingly, with the exception of the few Ti-Mn samples prepared by jet polishing mentioned above. Milling was performed at a 15° incidence to the sample, with power settings of 6.0 V and 1.6 mA to perforation. Once a hole formed in the sample, the power was reduced to 3V and 0.5mA for an additional milling time of 30 minutes in order to remove amorphous material from the sample edge and leave a very thin sample area suitable for HREM.

#### **2.8. Transmission electron microscopy – TEM**

TEM was used for the majority of characterization and analysis in this study. A JEOL JEM-4000FX microscope operating at 300KeV was used. A combination of many techniques was required to understand the microstructure and deformation mechanisms of Ti-6.0Mn and Ti-8.1V, including selected area diffraction (SAD), bright field imaging, dark field imaging, and high resolution microscopy. A complete description of these techniques is beyond the scope of this chapter, but an excellent resource for various TEM techniques is [68]. Several important points must be kept in mind for the safety and operation of the instrument which are outlined in Appendix E.

The various deformation mechanisms were identified in several ways. Slip was identified by dark field imaging using three different g vectors, and  $g \bullet b$  analysis. In order to determine the dislocation line direction, the diffraction pattern must be oriented correctly with respect to the image. This rotation changes depending on the magnification of the image. The author performed this calibration during this investigation since the rotations were not known for the JEM-4000FX, using a specimen of epitaxial SiC on (100) Si specimen provided by Prof. Salamanca-Riba. This calibration is provided in Appendix F.

To determine if slip involving screw dislocations with a Burgers vector  $\vec{b} = 1/3\langle 11\bar{2}0 \rangle$  where gliding on basal or prism planes, bright and dark field images were taken along both  $\langle 11\bar{2}0 \rangle_{\alpha}$  and  $[0001]_{\alpha}$  zone axes and tilted slightly to determine if the dislocation lines visible were single dislocations or stacks of dislocations on single planes in projection. This was necessary because 'a' type screw dislocations in hexagonal crystals can glide on the basal or prism slip planes.

To identify twins in the  $\alpha$  phase selected area diffraction patterns (SADPs) were taken from the matrix, the twin/matrix interface, and where possible the twin. In general to identify the type of twin the specimen must be tilted along a zone axis that contains the g vector for the twin plane common to both the twin and the matrix. For the  $\left\{10\overline{1}1\right\rangle\left\langle\overline{1}012\right\rangle$  and  $\left\{10\overline{1}2\right\rangle\left\langle\overline{1}011\right\rangle$  twins observed in this investigation it was required to tilt along a  $\langle 11\overline{2}0 \rangle_{\alpha}$  zone axis. In the case of  $\{10\overline{1}1\}$  twins there is a rotation of ~122° between the [0001] planes of the twin and matrix, and for  $\{10\overline{1}2\}$  twins the rotation will be ~94° [18]. The exact rotation depends of the c/a ratio of the  $\alpha$  phase. The spots in the interface diffraction pattern will be mirrored across the g vector of the twin plane. Since there are three independent  $\langle 11\overline{2}0 \rangle_{\alpha}$  directions, and the twin plane contains only one of these directions, for each twin there is only a 1/3 chance that the correct zone axis for identification will be attainable, as tilting 60° to the next  $\langle 11\overline{2}0 \rangle_{\alpha}$  axis is impossible due to limits of the TEM specimen holder. In even more cases the alignment of the  $\alpha$  grain where the twin is visible is oriented close to the  $[0001]_{\alpha}$  zone axis, where a tilt of ~90° would be required. Therefore identification of any given twin is extremely difficult, and some twins could not be identified due to the tilt limits of the specimen holder. Persistence and patience must be used during this process. During the course of this investigation it became apparent that twins were misidentified in a previous study [57]. To avoid misidentification in future studies a short guide to twin identification in  $\alpha$ titanium has been prepared in Appendix G.

As discussed briefly in the introduction, there have been several type of stress induced martensite reported in titanium, alloys. To identify which type of martensite was

present, the martensite selected area diffraction patterns were indexed and measured. The crystal structure was identified as hexagonal, and the Burgers orientation relationship  $[0001]_{\alpha'}$  //{ $\{110\}_{\beta}$ , which exists between the martensite and the  $\beta$  phase, was obeyed. This was evidence for stress induced hexagonal martensite as opposed to the orthorhombic martensite. The stress induced hexagonal martensite can form in six orientations in any given  $\beta$  grain with  $[0001]_{\alpha'}$  //{ $\{110\}_{\beta}$ , so the martensite may not be oriented along an equivalent  $\beta$  zone axis to the one aligned with the beam, so identification of an individual martensite plate may be difficult.

It should be noted at this time that undeformed material was examined to insure that the deformation mechanisms in the deformed material were the result of testing and not TEM specimen preparation. Also, the frequency of twins and martensite is quite low, therefore slow and careful surveying of each TEM specimen was necessary to find areas with twins and martensite for analysis. The objective lens aperture must be used in order to distinguish these plates from the matrix, and even then the contrast can be faint.

### 2.9. High resolution electron microscopy – HREM

High resolution electron microscopy (HREM) was used to examine the interface structure between the  $\alpha$ ,  $\beta$ , and  $\omega$  phases of the two-phase Ti-6.0Mn and Ti-8.1V alloys with Widmanstätten microstructure. A JEOL JEM-4000FX microscope operating at 300KeV was used. The microscope must be very well aligned, including the objective lens astigmatism, in order to obtain high quality HREM images. The details of the alignment procedure are too lengthy to include here, but can be found in the 4000FX operating manual. In most cases the microscope maintains its alignment well, and only

slight realignment is necessary. The alignment information, given as the current through various lenses, should be recorded when the microscope is aligned well in the case that a gross misalignment occurs.

In order to record images of the  $\alpha$ - $\beta$  and  $\alpha$ - $\omega$  interfaces, the following procedure was used. Due to the Burgers orientation relationship between phases, the sample can be oriented in such a way that the beam direction will be parallel with the interface plane. Because the habit plane is perpendicular to both the basal plane of the  $\alpha$  phase, and the (110) plane of the  $\beta$  phase, the sample must be tilted so that the  $[0001]_{\alpha}$  and  $[110]_{\beta}$  zone axis are aligned with the electron beam. Diffraction patterns should be taken from each side of the interface, and should appear symmetrical in intensity. Even a slight tilt off of the above zone axes will result in a HREM image that is poorly defined at the interface due to overlap of the  $\alpha$  and  $\beta$  phases, and the lattice fringes will be stronger in one direction. The diffraction patterns of each phase should be confirmed at the actual site of HREM imaging using the smallest selected area aperture to insure a perfect tilt, as the sample edges where the specimen is thinnest may be bent slightly in relation to areas further away from the edges. In order to examine the martensite/ $\alpha$  interface (which is discussed in Chapter 7) as well as the  $\alpha/\beta$  interface it was also desirable to examine the interface from the  $[1\overline{2}10]_{\alpha}//[1\overline{1}1]_{\beta}$  zone axes. This direction is only 11° away from the  $\alpha/\beta$  interface plane, and the beam will be parallel to the martensite/ $\alpha$  interface plane.

Since the material is polycrystalline, some areas of each specimen will be better aligned than others with the beam when the sample holder is at 0° tilt and azimuth. The best results are obtained when tilting is kept to a minimum, since the cross section that electrons must pass through increases with tilting. Therefore, effort should be taken to

find a well thinned region of the sample, where the  $\alpha$  and  $\beta$  phases have equal thickness, and the sample is closely aligned with the required zone axes with little tilting.

In order to form a HREM, or lattice fringe, image of a material the objective lens aperture must be used to limit the number of beams used for the image. Highest contrast is obtained when all the first order beams are used to form the image, and the remaining beams are excluded by the objective lens aperture. All first order beams must be used to image precise atomic location. When attempting to image the  $\beta$  phase from the  $[110]_{\beta}$  zone axis difficulties were encountered due to the small lattice spacing. The lattice spacing of the  $\{002\}_{\beta}$  planes is only 1.63 Å, and the  $\{112\}_{\beta}$  planes have a lattice spacing of only 1.33 Å, therefore the selected area diffraction spot distances are quite large. The first objective lens aperture is much too large, and includes too many beams for a high contrast HREM image, but the second objective lens aperture is too small to include the  $\{002\}_{\beta}$  and  $\{112\}_{\beta}$  beams. To overcome this problem, the objective lens aperture was raised very close to the sample such that the above beams are included in the HREM image with the second aperture. This can only be done once the sample has been tilted into perfect alignment with the beam, as further tilting once the aperture has been moved will result in the specimen holder contacting the objective lens aperture. Once the HREM images have been taken, the objective lens aperture must be returned to its original height to prevent damage. It should be noted that even with this technique, the lattice spacing of these planes approached the resolution limit of the microscope. It was nearly impossible to resolve a clear high resolution image of the  $\beta$  phase, and lattice fringes in one direction were the usual result.

High resolution images were normally recorded at magnifications of 400 kx, although in some areas the interference fringes of planes with small lattice spacing were difficult to see, and images were recorded with good results at magnifications of 500-600 kx. At these high magnifications care should be used that microscope is focused carefully to the optimum defocus condition, and that no vibration, including sound, moves the specimen, which will blur the image. HREM is best performed in the evenings when the building is quiet. Negatives were scanned at either 2400 or 7200 dpi using an Epson Perfection 1200 photo scanner with backlighting attachment.

## 2.10. Calculation of resolved shear stresses

In order to understand the effect of interactions between  $\alpha$  and  $\beta$  phase deformation mechanisms, the magnitude of resolved shear stress that the observed  $\alpha$ phase deformation mechanisms placed on the shear systems for twinning and the  $\beta \rightarrow \alpha'$ and  $\omega \rightarrow \alpha'$  transformations were calculated for all possible combinations of observed deformation mechanisms. Due to the Burgers orientation relationship between the  $\alpha$  and  $\beta$ phases and the  $\omega$  and  $\beta$  phases, the orientation of the various directions and planes are fixed in relation to one another within the alloy system.

The 4-index coordinate systems for the hexagonal  $\alpha$  and  $\omega$  phases were first transformed to a 3-coordiante Cartesian system by the equations in Appendix H. Transformation matrices were calculated to transform directions in the  $\alpha$  and  $\omega$  phases into parallel directions in the  $\beta$  phase [69]. The equivalent directions needed for the calculation of these matrices are given by the Burgers orientation relationship between  $\alpha$  and  $\beta$  and the orientation relationships for each of the four orientations of  $\omega$  phase and  $\beta$ 

[17, 41]. These transformation matrices are given in Appendix H. Directions and planes important for calculating resolved shear stresses, such as slip planes, Burgers vectors, twinning planes and directions, and planes and directions important for the  $\omega \rightarrow \alpha'$ transformation were then converted to equivalent directions in the  $\beta$  phase, which are presented in Appendix I. The result is a common Cartesian coordinate system to perform calculations of resolved shear stresses between the  $\alpha$ ,  $\beta$ , and  $\omega$  phases.

Within this coordinate system the magnitude of resolved shear stress from deformation products in the  $\alpha$  phase were calculated. The shear stresses for three basal slip systems, three prism slip systems, and six twinning shear systems for each of the  $\{10\overline{1}1\rangle\langle\overline{1}012\rangle$  and  $\{10\overline{1}2\rangle\langle\overline{1}011\rangle$  twins were resolved onto the 12 possible shear systems for twinning and 24 shear systems for the  $\beta \rightarrow \alpha'$  stress induced transformation. Additionally, the resolved shear stress was calculated for the above  $\alpha$  phase deformation features onto the shear systems for the  $\omega \rightarrow \alpha'$  transformation. The magnitude of these resolved shear stresses was calculated for every combination of above deformation products in an Microsoft Excel spreadsheet using the equations for vector transformation given below [70]:

$$\sigma_{x'y'} = \alpha_1 \alpha_2 \sigma_{xx} + \beta_1 \beta_2 \sigma_{yy} + \gamma_1 \gamma_2 \sigma_{zz} + (\alpha_1 \beta_2 + \alpha_2 \beta_1) \sigma_{xy} + (\beta_1 \gamma_2 + \beta_2 \gamma_1) \sigma_{yz} + (\alpha_1 \gamma_2 + \alpha_2 \gamma_1) \sigma_{xz}$$

$$(2.1)$$

where:

and [ijk] is the original vector and [i'j'k'] is the vector on which the stress is resolved. The complete results of these calculations are presented in Appendix J.

 $\begin{aligned} \alpha_1 &= i' \bullet i \quad \alpha_2 &= j' \bullet i \\ \beta_1 &= i' \bullet j \quad \beta_2 &= j' \bullet j \end{aligned}$ 

 $\gamma_1 = i' \bullet k \quad \gamma_2 = j' \bullet k$ 

# **Chapter 3**

# Influence of the Second Phase on the Room Temperature Deformation Mechanisms of α-β Titanium Alloys with Widmanstätten Microstructure

# **Part 1: Tensile Deformation**

The effects of  $\alpha$  and  $\beta$  phase interactions on the tensile deformation behavior of  $\alpha+\beta$  titanium alloys with Widmanstätten microstructures were studied using Ti-6.0wt%Mn and Ti-8.1wt%V as the model two-phase systems. It was found that when the  $\alpha$  phase is present as thin (<10µm) plates in the  $\alpha+\beta$  alloys, significant twinning occurs. No significant twinning was observed in single-phase alloys with the same chemistry and similar grain size. Additionally, the  $\beta$  phase of Ti-8.1V deforms by stress induced hexagonal martensite ( $\alpha'$ ), while only twinning occurs in the single-phase  $\beta$  alloy with the same chemistry. This behavior is modeled in terms of a number of factors including elastic interaction stresses between the  $\alpha$  and  $\beta$  phases, coherency between the  $\alpha$  phase and hexagonal martensite, and  $\beta$  phase stability.

# **3.1. Introduction**

Numerous studies have been conducted on the low temperature tensile deformation mechanisms of single-phase  $\alpha$  and  $\beta$  alloys, as well as two-phase  $\alpha + \beta$ titanium alloys. The deformation mechanisms of single-phase  $\alpha$  titanium alloys are fine slip, coarse slip, and twinning [71]. The deformation mechanisms of single-phase  $\beta$  alloys include fine slip, coarse slip, twinning, and the formation of stress induced martensite or stress induced  $\omega$  phase [17, 71]. Two-phase  $\alpha+\beta$  alloys can deform by the mechanisms listed above for the individual phases, as well as by interphase-interface sliding [49].

In regard to the Widmanstätten  $\alpha+\beta$  microstructures it is well known that a Burgers orientation relationship exists between the  $\alpha$  and  $\beta$  phases, i.e. slip systems in  $\alpha$ and  $\beta$  are parallel [1, 36, 41, 72-74]. Slip can be transmitted across the  $\alpha$ - $\beta$  interface from a slip system in one phase to another in the adjacent phase [48]. However, there are no systematic studies that relate the deformation mechanisms of individual phases to those of the two-phase materials. This is not a simple extension of the behavior of single-phase materials because of interactions between the phases. These include elastic interaction between phases, localized stress from deformation products in adjacent phases, and  $\alpha$ phase templating due to the Burgers orientation relationships mentioned above. In this regard the present investigation addresses the effect of the  $\alpha$  phase on the deformation mechanisms of the  $\beta$  phase, and vice-versa, in  $\alpha - \beta$  Widmanstätten titanium alloys and their relationship to the behavior of the single phase  $\alpha$  and  $\beta$  alloys.

# **3.2.** Experimental procedure

Single and two-phase Ti-Mn and Ti-V alloys were selected such that the chemistry of the single phase  $\alpha$  and  $\beta$  alloys would match the component  $\alpha$  and  $\beta$  phases of the two-phase alloys. The model two-phase alloys are Ti-6.0wt%Mn and Ti-8.1V. A complete listing of chemistry of the single and two-phase alloys , along with the Ti-Mn and Ti-V phase diagrams, are given in Appendix A. Tensile testing and SEM analysis of these alloys have been performed during the course of previous investigations [25, 27-29,

75]. The experimental work of this investigation is primarily directed at TEM studies and modeling the effect of microstructure on the deformation mechanisms of two-phase alloys.

The two-phase alloys were heat treated according to the procedure given in Chapter 2.1, and tensile specimens were prepared from these alloys by the procedure given in Chapter 2.2. Gold fiducial lines were affixed to the surface of the gage section by the procedure given in Chapter 2.3.

Ambient temperature tensile tests were performed at a strain rate of  $3.28 \times 10^{-5}$  per second to a total strain of 3%. Optical and SEM micrographs were taken in the same area of the polished gage sections before and after testing to record deformation products, including interphase interface sliding, stress induced plate formation, and slip [75].

Following tensile testing, TEM specimens were prepared from the gage length (deformed material) and from the grip section (un-deformed material) using the procedure given in Chapter 2.7. Electro-chemically thinned Ti-6.0Mn TEM samples were also analyzed to assess the degree of  $\omega$  formation in the  $\beta$  phase without the possibility of inducing thermal  $\omega$  phase by heating during ion milling. This is of no concern with Ti-8.1V, as athermal  $\omega$  phase is already present in the  $\beta$  phase. A JEOL JEM-4000FX transmission electron microscope, operating at 300 KeV, was used to obtain the required diffraction patterns and images for this study.

# 3.3. Results

The tensile deformation mechanisms of the single-phase  $\alpha$  and  $\beta$  alloys of the Ti-Mn and Ti-V systems have been explored during the course of previous studies [25,

27-29]. The single-phase  $\alpha$  alloys, Ti-0.4Mn and Ti-1.6V, deform by similar mechanisms. Ti-0.4Mn with large grain size (500 µm) has a yield stress (YS) of 262 MPa, and deforms in tension by a combination of slip and the formation of stress induced  $\{10\overline{1}1\}$  twins, whereas small grained specimens (45 µm) have a YS of 235 MPa and deform solely by fine slip [27]. The twins were previously identified as  $\{10\overline{1}2\}$  type, but analysis of the selected area diffraction pattern (SADP) in the current investigation reveals that the original analysis was in error. The small increase of the YS with increased grain size was attributed to the higher heat treatment temperature used to obtain a large grain size, which may have resulted in a lower number of dislocations available for slip. Similarly, the  $\alpha$ alloy Ti-1.6V with large grain size (226µm) has a YS of 344 MPa and deforms by a combination of slip and  $\{10\overline{1}1\}$  twinning, whereas the same alloy with small grain size (52-62 µm) has a YS of 334 MPa and deforms only by slip [29].

The deformation mechanisms of the single-phase  $\beta$  alloys, Ti-13.0Mn and Ti-14.8V, are dependent on their stability as well as grain size. The stability of the  $\beta$  alloys is given by molybdenum equivalency (MoE) [4]. It is a relative measure of the stability of a  $\beta$  titanium alloy with any number of stabilizing elements as compared to a Ti-Mo alloy with equivalent stability. The MoE has been calculated as 19.9 for Ti-13.0Mn and 9.9 for Ti-14.8V [61], therefore the stability of Ti-13.0Mn is much higher than that of Ti-14.8V. Ti-13.0Mn has a YS of 940 MPa, and deforms in tension solely by coarse and wavy slip, even in specimens with relatively large grain sizes (200 µm) [27]. In contrast, the Ti-14.8V alloy with large grains (350 µm) has a YS of 774 MPa, and deforms by slip and the formation of stress induced {332}(113) twins [25, 28]. These twins contained two orientations of the  $\omega$  phase. Specimens with smaller grain sizes (18-35  $\mu$ m) had YS values ranging from 876 to 900 MPa, and deformed solely by slip [28].

The two-phase Ti-6.0Mn and Ti-8.1V alloys were tensile tested at ambient temperature at a strain rate of  $3.28 \times 10^{-5}$  / second to a total strain of 3% [27, 75]. Figure 3.1 shows the tensile curves of both alloys.



**Figure 3.1.** Tensile curves of Ti-6.0wt%Mn and Ti-8.1wt%V alloys, tested at a strain rate of  $3.28 \times 10^{-5}$  / second to 3% total strain [27, 75].

Ti-6.0Mn exhibits a slightly higher YS than Ti-8.1V, while the elastic modulus of each alloy is approximately equal. Ti-6.0Mn yields at 623 MPa and strain hardens, while Ti-8.1V yields at 597 MPa and shows no significant strain hardening [27, 75].

Tensile deformation of Ti-6.0Mn is due primarily to fine slip in the  $\alpha$  phase, as well as limited  $\alpha$  phase twin formation and interphase interface sliding. SEM observations of the sample surface following testing revealed a small number of stress

induced plates in the  $\alpha$  phase. TEM analysis of Ti-6.0Mn following tensile testing revealed extensive slip as well as infrequent twins in the  $\alpha$  phase. Micrographs of these twins and the accompanying diffraction patterns are shown below in Figures 3.2 and 3.3. Twins were identified as  $\{10\overline{1}2\}$  type. Slip occurred primarily with screw dislocations on prism planes with  $\vec{b} = 1/3\langle 11\overline{2}0\rangle$ . Figure 3.4 shows a dark field TEM micrograph with slip occurring on three prism planes simultaneously. No slip or stress induced plates were observed in the  $\beta$  phase of Ti-6.0Mn.



**Figure 3.2**. TEM micrograph showing two parallel twins (indicated by arrows) in the  $\alpha$  phase of  $\alpha$ - $\beta$  Ti-6.0Mn following tensile deformation. No significant deformation features were observed in the  $\beta$  phase.



**Figure 3.3.** TEM image of twin in the  $\alpha$  phase of  $\alpha$ - $\beta$  Ti-6.0Mn from Figure 3.2. To the right are selected area diffraction patterns taken (a) Outside the twin and (b) Across the twin boundary. Zone axis is close to  $[11\overline{2}0]_{\alpha}$ . The twin is  $\{10\overline{1}2\}$  type.



**Figure 3.4.** Dark field TEM micrograph of  $\alpha$ - $\beta$  Ti-6.0Mn showing slip on prism planes in the  $\alpha$  phase. Dislocations are 'a' type screw with  $\vec{b} = 1/3 \langle 11\overline{2}0 \rangle$ .

In contrast to Ti-6.0Mn, SEM analysis of Ti-8.1V revealed coarse deformation products spanning many  $\alpha$  and  $\beta$  grains in the tensile specimens. These are absent in the Ti-6.0Mn specimens. The difference in deformation mechanisms is evident in the SEM micrographs of the gage sections Ti-6.0Mn and Ti-8.1V following tensile testing shown in Figures 3.5 and 3.6. These deformation products were observed to travel across the width of the grains as shown in Figure 3.5, or along the length of the  $\alpha$  and  $\beta$  grains, depending on the specimen area. Initially the deformation features in Ti-8.1V were reported to be coarse slip crossing through the phase boundaries of the alloy [27, 75]. However, the present investigation revealed that these deformation products are not coarse slip lines.



**Figure 3.5.** SEM micrograph of (a) Ti-6.0Mn and (b) Ti-8.1V alloy following tensile deformation to 3% total strain. Arrows indicate coarse deformation products in the  $\alpha$  phase of both alloys, whereas coarse deformation products span the  $\alpha$  and  $\beta$  phases of Ti-8.1V. Gold grid lines are applied to the sample surface by sputtering [27, 75].



**Figure 3.6.** SEM micrograph an area magnified from Figure 3.5 of Ti-8.1V alloy following tensile deformation to 3% total strain. In this region the deformation products zigzag across the width of  $\alpha$  (light) and  $\beta$  (dark) grains, as indicated by arrows [27, 75].

TEM analysis of the Ti-8.1V specimens revealed that the coarse lines visible in SEM micrographs are actually a combination of stress induced hexagonal martensite plates ( $\alpha$ ') in the  $\beta$  phase and either twins or coarse slip in the  $\alpha$  phase. This is the first time twinning in the  $\alpha$  phase has been reported in association with stress induced martensite in the  $\beta$  phase of a two-phase titanium alloy. It was often observed that plates of martensite in the  $\beta$  phase alternated with twins or coarse slip in the  $\alpha$  phase over a distance of many  $\alpha$  and  $\beta$  platelets. The TEM bright field micrograph shown in Figure 3.7 shows a series of martensite plates in the  $\beta$  phase and twins in the  $\alpha$  phase. Note the similarity of the deformation morphology to those in the SEM micrograph Figure 3.6.



**Figure 3.7.** TEM micrograph of Ti-8.1V following tensile deformation showing stressinduced hexagonal martensite plates in the  $\beta$  phase and twins in the  $\alpha$  phase, alternating over large numbers of  $\alpha$  and  $\beta$  grains. This identification is based on analysis of similar areas in the specimen. Identification could not be made in this area due to specimen orientation.

Both  $\{10\overline{1}\}$  and  $\{10\overline{1}2\}$  twins were observed in the  $\alpha$  phase of Ti-8.1V. Figure 3.8

shows a  $\{10\overline{1}\}$  type twin, and Figure 3.9 shows two  $\{10\overline{1}2\}$  twins in the  $\alpha$  phase of Ti-

8.1V. The accompanying diffraction patterns were taken in the parent  $\alpha$ , just inside the

twin, and across the twin-matrix interface, respectively.



**Figure 3.8.** Bright field TEM micrograph of a  $\{10\overline{1}1\}$  type twin in the  $\alpha$  phase of Ti-8.1V. Selected area diffraction patterns taken: (a) Outside of twin in  $\alpha$  phase (b) Inside of twin (c) across twin/ $\alpha$  boundary. Zone axis is  $[11\overline{2}0]_{\alpha}$ .



**Figure 3.9.** Bright field TEM micrograph of two  $\{10\overline{1}2\}$  type twins in the  $\alpha$  phase of Ti-8.1V. Selected area diffraction patterns taken: (a) Outside of Twin 2 in  $\alpha$  phase (b) inside of Twin 2 (c) across Twin 2 /  $\alpha$  boundary. Zone axis is  $[11\overline{2}0]_{\alpha}$ .

Two types of stress induced martensite are common in metastable  $\beta$  alloys, the hexagonal martensite ( $\alpha'$ ) and orthorhombic ( $\alpha''$ ). Numerous micrographs and diffraction patterns were taken to confirm that the stress-induced plates in the  $\beta$  phase were indeed hexagonal martensite, of which a selection are presented below. The selected area diffraction patterns indicate a hexagonal martensite structure, and the Burgers orientation relationship was obeyed between the martensite and the  $\beta$  phase. Further, the martensite was not internally twinned and has a straight  $\alpha'/\beta$  interface, which suggests a  $\{334\}_{\rho}$  habit plane [35]. Although Bagariatskii [37] states that the orthorhombic martensite will form in Ti-V alloys with a V concentration over 9.4 wt% and in Ti-Mo alloys with a Mo concentration over 4.0wt%, these results conflict with the work of Koul and Breedis [32] who report stress induced hexagonal martensite in Ti-16.2wt%V and Ti-12.9wt%Mo alloys.

Figure 3.10 is a bright field TEM micrograph of three stress induced hexagonal martensite plates in the  $\beta$  phase and a  $\{10\overline{1}2\}$  twin in the  $\alpha$  phase of Ti-8.1V. The accompanying selected area diffraction patterns identify the twin, the stress induced martensite plate contacting the twin, the  $\alpha/\beta$  Burgers orientation relationship, and the  $\{10\overline{1}1\}$  twinned relationship between the  $\alpha'$  plate in the  $\beta$  phase and the adjacent  $\alpha$  phase, which is discussed further in Section 4.2.2 below. Figure 3.11 shows a martensite plate in the  $\beta$  phase, with accompanying selected area diffraction patterns taken outside the martensite plate along the  $(1\overline{1}1)$  zone axis of  $\beta$  and another across the martensite/ $\beta$  boundary that includes spots from the parallel  $(1\overline{1}1)_{\beta}$  and  $(1\overline{2}10)_{\alpha'}$  zone axes. A series of

parallel martensite plates with accompanying diffraction patterns, viewed along the  $(110)_{\beta}$  //(0001)<sub> $\alpha'$ </sub> zone axes, are shown in Figure 3.12. Multiple martensite plates in the  $\beta$  phase or twins in the  $\alpha$  phase are typically parallel, which is also shown in Figure 3.9 and 3.10. This is due to the alignment of the grain with a specific resolved shear stress in that region of the material. The stress induced martensite plates were completely transformed to  $\alpha'$ , with no remaining traces of  $\beta$  or  $\omega$  phase within the plates.



**Figure 3.10.** TEM bright field micrograph showing three hexagonal martensite plates ( $\alpha'$ ) in the  $\beta$  phase in combination with a  $\{10\overline{1}2\}$  type twin in the  $\alpha$  phase. The twin was classified specifically as  $(\overline{1}012)[10\overline{1}1]$ . Accompanying selected area diffraction patterns are from (a) the  $\alpha$  phase (b) the  $\alpha$  / twin interface (c) the  $\beta$  phase (d) the  $\alpha'_1$  /  $\beta$  interface (e) the  $\alpha'_1$  /  $\alpha$  interface showing a  $\{10\overline{1}1\}$  type twinned relationship. Zone axis is  $[1\overline{2}10]_{\alpha}$  // $[1\overline{1}1]_{\beta}$ .



**Figure 3.11.** Selected area diffraction patterns taken a) outside of the hexagonal martensite ( $\alpha'$ ) plate in the beta phase and b) across the interface of martensite plate and the beta phase of Ti-8.1V. Arrows indicate martensite. Zone axis is  $[1\overline{1}1]_{\beta}$  // $[1\overline{2}10]_{\alpha'}$ .



**Figure 3.12.** Stress induced hexagonal martensite ( $\alpha'$ ) plates (indicated by arrows) in the beta phase of Ti-8.1V. Diffraction patterns from (a) outside of the martensite plate in the beta phase, (b) inside the martensite plate, and (c) across the interface of martensite plate within the beta phase of Ti-8.1V. Zone axis is  $[110]_{\beta}$  // $[0001]_{\alpha'}$ .

Slip in the  $\alpha$  phase accompanies martensite formation in the  $\beta$  phase as often as twinning in the  $\alpha$  phase. Figure 3.13 shows a martensite plate in  $\beta$  spanning between two  $\alpha$  grains with coarse slip lines. The dislocations on one side of the martensite plate were found to be a combination of 'a' and 'c' type dislocations on prism slip planes, and 'a' type prism slip on the other. This was the only observed instance of slip with a Burgers vector  $\vec{b} = \langle 0001 \rangle$ . Every other instance of slip has Burgers vector  $\vec{b} = 1/3 \langle 11\overline{2}0 \rangle$ . A dark field image of the lower left area of Figure 3.13 is shown in Figure 3.14. Screw dislocations on prism planes are present throughout this  $\alpha$  grain. Prism slip with 'a' type Burgers vector and screw orientation is the predominant slip system in Ti-8.1V. Another example of slip in  $\alpha$  and martensite plates in  $\beta$  from a different area is shown in Figure 3.15. Slip was not observed in the  $\beta$  phase of Ti-8.1V.



**Figure 3.13.** Bright field TEM micrograph of martensite plates in the  $\beta$  phase and slip in the  $\alpha$  phase of Ti-8.1V. Prism slip at (a) and (b) is due to 'a' type dislocations. Prism slip at (c) is due to 'c' type dislocations. Area (d) is expanded in Figure 3.14.



**Figure 3.14.** Dark field image of  $\alpha$  plate from Figure 3.13. Screw dislocations are visible on both slip system A=  $(\overline{1} 100)\pm 1/3[1\overline{2}0]$  and slip system  $B = (10\overline{1}0)\pm 1/3[1\overline{2}10]$ . Arrows indicate projection of Burgers vector. Stress induced martensite (SIM) plate is indicated in the  $\beta$  phase.



**Figure 3.15.** Slip in  $\alpha$  phase of Ti-8.1wt%V adjacent to stress induced martensite plates in  $\beta$  phase (indicated by arrows).

These results clearly show that the deformation mechanisms in a two-phase alloy can differ from those of the single-phase alloys equivalent to either of the two component phases.

# 3.4. Discussion

Three phenomena occurred during tensile deformation of the two-phase Ti-6.0Mn and Ti-8.1V alloys that were unexpected when considering the deformation behavior of their component single-phase alloys. The first is twinning in small  $\alpha$  grains. The second phenomenon is deformation by stress induced martensite in the  $\beta$  phase of the two-phase alloy Ti-8.1V, when deformation in the matching single phase  $\beta$  alloy, Ti-14.8V, occurs by twinning. Finally, the third phenomenon is the formation of stress induced martensite in the  $\beta$  phase of Ti-8.1V but not in the  $\beta$  phase of Ti-6.0Mn. In all of the above cases the Burgers orientation relationship illustrated in Figure 3.16 plays an important role in the interactions between the  $\alpha$  and  $\beta$  phases in alloys with a Widmanstätten microstructure. A model which incorporates influences of the  $\beta$  phase on the deformation behavior of the  $\alpha$  phase, as well as influences of  $\alpha$  on the deformation of the  $\beta$  phase, is presented below.



**Figure 3.16.** Illustration of the  $[1\overline{2}10]_{\alpha}(0001)_{\alpha}//[1\overline{1}1]_{\beta}(1\overline{1}1)_{\beta}$  Burgers orientation relationship between the  $\alpha$  and  $\beta$  phases in an  $\alpha$ - $\beta$  titanium alloy with Widmanstätten microstructure. Diagram shows the  $(0001)_{\alpha}$  and  $(110)_{\beta}$  planes. The interface plane is  $(\overline{5}140)_{\alpha}//(\overline{3}34)_{\beta}$ , which is normal to the  $(0001)_{\alpha}$  and  $(110)_{\beta}$  planes and is indicated a trace in each phase. Figure is drawn to scale.

# 3.4.1 Twins in small $\alpha$ grains of $\alpha$ - $\beta$ alloys

The first phenomenon of interest is twinning in the  $\alpha$  phase of both Ti-6.0Mn and Ti-8.1V. Previous studies of the single-phase  $\alpha$  alloys of each system (Ti-0.4wt%Mn and Ti-1.6wt%V) showed significant twin formation only in samples with large (>62 $\mu$ m) grain sizes [27, 29]. The average width of the  $\alpha$  platelets in the Ti-8.1V and Ti-6.0Mn

alloys is less then 10 $\mu$ m, with many grains having widths less than 1  $\mu$ m. The relationship between grain size and the required stress for twin formation,  $\sigma_{\rm T}$ , is given as [26]:

$$\sigma_{\rm T} = \sigma_{\rm T0} + k_{\rm T} d^{-1/2} \tag{3.1}$$

where  $\sigma_{T0}$  is the stress required to initiate a twin,  $k_T$  is the Hall-Petch slope, and *d* is the grain size. The occurrence of twins in the small  $\alpha$  platelets of Ti-8.1V and Ti-6.0Mn is therefore unlikely unless additional contributions are made to the twinning stress, such as those due to elastic interaction stresses between the stronger but lower modulus  $\beta$  phase and the softer but higher modulus  $\alpha$  phase. The contribution of elastic interaction stress to the twinning stress is explained below.

### **3.4.1.1 Elastic interaction stresses**

Elastic interaction stresses between the  $\alpha$  and  $\beta$  phases in titanium alloys may be responsible for a number of phenomena, including twin formation in the small-grained  $\alpha$ phase and stress induced martensite formation in the  $\beta$  phase. Elastic interaction stresses arise due to differences in the elastic properties of two ductile phases which are constrained at an interface. The well defined interface between the phases constrains the  $\alpha$  and  $\beta$  phases, such that the strain in each phase at the interface must be equal. If one considers the Cartesian coordinate system *xyz* such that x and z lie in the interface plane, then the continuity of strain across the interface is expressed by:

$$e_{xx}^{\alpha} = e_{xx}^{\beta}, \ e_{zz}^{\alpha} = e_{zz}^{\beta}, \ e_{zx}^{\alpha} = e_{zx}^{\beta}$$
(3.2)

The elastic interaction stresses have been analyzed for two-phase titanium systems in several studies [41, 73, 76-78]. Ankem and Margolin [41] analyzed the

interaction stresses in two-phase titanium alloys with Widmanstätten microstructure for numerous parallel  $\alpha$  and  $\beta$  slip systems (they referred to parallel planes and directions as slip systems even though slip may not actually occur). Figure 3.17 below shows the generalized shear stress – shear strain curves of the individual phases of an  $\alpha$ - $\beta$  alloy for the basal slip system in  $\alpha$  and the parallel slip system in  $\beta$ . The yield strength of the  $\alpha$ phase is lower than that of the  $\beta$  phase, and the elastic modulus of the  $\beta$  phase is lower than that of the  $\alpha$  phase. The same properties exist for the  $\alpha$  and  $\beta$  phases of both Ti-6.0Mn and Ti-8.1V.

An analysis of Figure 3.17 shows that initially, at a given applied stress on the two-phase alloy, the stress and strain of the  $\alpha$  phase will be increased in excess of the applied stress level, while the stress on the  $\beta$  phase will be decreased in order to maintain an equivalent strain on each phase at the interface. Therefore, the  $\alpha$  phase in the two-phase alloy can deform plastically at applied stress levels that would normally result only in elastic deformation in a single-phase alloy. The elastic interaction stresses on the  $\alpha$  phase of both Ti-6.0Mn and Ti-8.1V are similar, so the result of the additional stress is the formation of twins in the small  $\alpha$  platelets of both alloys. This is observed experimentally. Slip can also occur in the  $\alpha$  phases of both these alloys. The occurrence of slip or twinning in an  $\alpha$  platelet is determined by the orientation of the  $\alpha$  grain to the applied stress axis and if mobile dislocations are available.



**Figure 3.17.** Elastic interaction stress on  $\alpha$  and  $\beta$  phases of a titanium alloy. The interaction of the stronger, lower modulus  $\beta$  phase with the  $\alpha$  phase initially increases the stress in  $\alpha$ . Once significant plastic deformation occurs in  $\alpha$ , elasto-plastic interaction stresses act on the  $\beta$  phase [41].

# 3.4.2 Differing $\beta$ phase deformation mechanisms between single and two-phase Ti-V alloys

The second phenomenon of interest is the formation of stress induced hexagonal martensite plates in the  $\beta$  phase of Ti-8.1V in contrast to the formation of {332} type twins in the same  $\beta$  phase when present as a single phase, Ti-14.8V. In order for stress induced martensite to occur in place of twinning, the relative activation energy must be lowered relative to twinning, or additional stresses which aid the martensite transformation must be applied. The difference between single and two-phase alloy

behavior can be explained by elasto-plastic interaction stresses,  $\alpha$  phase templating and resolved shear stresses from  $\alpha$  phase deformation products.

#### **3.4.2.1 Elasto-plastic interaction stresses**

Interaction stresses can be placed on the  $\beta$  phase as well as the  $\alpha$  phase. These stresses can be considered elasto-plastic, since one phase ( $\beta$ ) is deforming elastically while the other ( $\alpha$ ) is plastically deforming. A further examination of Figure 3.17 shows that after significant plastic deformation occurs in the  $\alpha$  phase, and the strain on the alloy extends to the right of the intersection of the  $\alpha$  and  $\beta$  stress-strain curves, interaction stresses are now placed on the  $\beta$  phase. The  $\beta$  phase must now constrain the plastically deforming  $\alpha$  phase, which increases the stress in the  $\beta$  phase while lowering the stress on  $\alpha$ . The additional stress acting on the  $\beta$  phase due to elastic interaction with the  $\alpha$  phase contributes to  $\beta$  phase deformation, including the formation of stress-induced martensite in the  $\beta$  phase of Ti-8.1V.

#### 3.4.2.2 α phase as a template for stress induced martensite

The activation energy for stress induced martensite may be lowered in a twophase titanium alloy by the  $\alpha$  phase acting as a template for the formation of stress induced martensite. The well defined orientation relationship between the  $\alpha$  and  $\beta$  phases, and between the  $\beta$  phase and hexagonal martensite, leads to a  $\{10\overline{1}1\}$  twin relationship between the  $\alpha$  phase and a martensite plate within the  $\beta$  phase. TEM selected area diffraction patterns confirm that a martensite plate and an adjacent  $\alpha$  grain share a common  $\{10\overline{1}1\}$  type plane. This relationship is visible in the selected area diffraction

patterns from the  $\alpha$  phase,  $\alpha'/\beta$  interface, and  $\alpha'/\alpha$  interface diffraction patterns in Figure 3.10 (a,d,e).

There is no such relationship between the  $\alpha$  phase and a {332} type twin in  $\beta$ . The common twin plane between the  $\alpha$  phase and hexagonal martensite may lower the nucleation energy of martensite formation compared to the activation energy required for twinning in a two-phase alloy. This nucleation template does not exist in the single-phase  $\beta$  alloy with identical chemistry and stability, therefore twins are the preferred deformation product.

## 3.4.2.3 Alignment of $\alpha$ and $\beta$ slip and shear systems

In order to facilitate the stress induced martensitic transformation by overcoming the activation energy, additional stresses can be added to the externally applied stress. The strain field due to dislocations, and presumably twins, can contribute to the stress required to nucleate stress induced martensite [79, 80]. The additional energy provided by these deformation features lowers the nucleation energy barrier for martensite formation.

The shear stress due to either slip or twinning in the  $\alpha$  phase acts in varying degrees on the shear systems of twinning or the  $\beta \rightarrow \alpha'$  transformation systems in the  $\beta$  phase. In two-phase titanium alloys with Widmanstätten microstructure, the Burgers relationship between the  $\alpha$  and  $\beta$  phases results in an alignment of slip or shear systems in the  $\alpha$  phase with shear systems in the  $\beta$  phase. The degree to which slip or twin systems in the  $\alpha$  phase act on either twin or martensite shear systems in the  $\beta$  phase helps to determine which deformation product is more likely to occur during tensile deformation.

The magnitude of resolved shear stresses from slip or twinning in the  $\alpha$  phase which act on the shear systems for either twinning or stress induced martensite in the  $\beta$ phase were calculated for all possible combinations of observed slip and twins in the  $\alpha$ phase and martensite and twins in the  $\beta$  phase. The shear systems for twins in the  $\beta$  phase of the Ti-V system are the type  $\{332\}\langle113\rangle$ . In the case of stress induced hexagonal martensite, Otte [74] proposed that the shear systems involved in the  $\beta$  to  $\alpha'$ transformation are:

$$[111]_{\beta} (11\overline{2})_{\beta} \equiv [2\overline{1}\overline{1}3]_{\alpha'} (\overline{2}112)_{\alpha'} \quad \text{and}$$
$$[111]_{\beta} (\overline{1}01)_{\beta} \equiv [2\overline{1}\overline{1}3]_{\alpha'} (\overline{1}011)_{\alpha'}$$

The observed deformation products in the  $\alpha$  phase are predominately 'a' type prism slip with Burgers vectors  $1/3\langle 11\overline{2}0\rangle$ , and both  $\{10\overline{1}1\}$  and  $\{10\overline{1}2\}$  twins.

Additionally, the resolved stress from 'a' type basal slip was calculated because of the possibility of cross slip within the  $\alpha$  phase in titanium alloys.

The resolved shear stress from prism slip in the  $\alpha$  phase acts strongly on both the twin and martensite shear systems in the  $\beta$  phase, with separate slip systems showing a maximum of 100% resolved shear stress onto the respective shear systems. Although prism slip was identified in the  $\alpha$  phase of both of the model alloys, it is possible for 'a' type screw dislocations to slip on the basal planes in titanium alloys. Therefore calculations of the resolved shear stresses from basal slip were also performed. The calculations for basal slip in  $\alpha$  show a significant amount of resolved shear stress (>85%) on several martensite shear systems from two of the three independent slip systems, but the greatest resolved shear stress on a twin shear system is only 81% from a single slip system. Therefore, the shear stress from slip in the  $\alpha$  phase could contribute to the shear
stress for martensite. Both the martensite and twin shear systems would be acted on approximately equally in the case of prism slip, but basal slip would clearly favor the formation of martensite.

Calculations of the resolved shear stresses from  $\{10\overline{1}1\}$  and  $\{10\overline{1}2\}$  type twins in the  $\alpha$  phase on the shear systems of twinning and stress induced martensite in the  $\beta$ phases indicate that generally that  $\alpha$  phase twins act to a much greater degree on martensite formation than twinning. Four of six  $\{10\overline{1}1\}$  twins resolve more than 94% of their shear stress on martensite shear systems, while only two of these same twins resolve a maximum of 91% shear stress on  $\beta$  twinning systems. The two  $\{10\overline{1}1\}$   $\alpha$  twins that resolve more stress onto  $\beta$  twin systems have a resolved shear stress magnitude of only 73%, compared with stresses of 62% on  $\beta \rightarrow \alpha'$  systems. Similarly, the contribution of shear stress from  $\{10\overline{1}2\}$  a phase twins acts more strongly on martensite transformation systems rather than twin shear systems. Four of six independent  $\{10\overline{1}2\}\alpha$  twins resolve only 75-80% of shear stress onto twin shear systems, while the same twins resolve 85-89% of shear stress onto martensite shear systems. The remaining two  $\{10\overline{1}2\}\alpha$  twins resolve only 54% of their yield stress onto twin shear systems compared to 50% onto martensite shear systems. For the greatest magnitudes of resolved stress the formation of stress induced martensite is favored due to the contributions of shear stress from  $\{10\overline{1}1\}$ twinning in the  $\alpha$  phase. For  $\alpha$  twins that act to a greater extent on  $\beta$  phase twin systems, the magnitude of resolved stresses are small, and thus do not contribute greatly to the activation of twins.

Table 3.1 shows a comparison of the maximum shear stresses imposed from basal slip, prism slip, and twins in the  $\alpha$  phase on either twins or stress induced martensite in the  $\beta$  phase. Complete tables of the calculated shear stress values are available in Appendix J. It is clear that the maximum resolved shear stresses from slip and twinning in the  $\alpha$  phase are greater on the shear systems for martensite formation rather than twinning.

**Table 3.1.** Maximum resolved shear stresses from  $\alpha$  deformation mechanisms acting on stress induced hexagonal martensite and  $\{332\}\langle 113 \rangle$  twin shear systems.

	α Deformation Mechanism			
β Shear System	Prism Slip	Basal Slip	$\{10\overline{1}1\}$ Twin	$\{10\overline{1}2\}$ Twin
$\{332\}\langle113\rangle$ Twin	1	0.81	0.91	0.80
Martensite - $\alpha$ '	1	1	0.95	0.89

The additional stresses from slip and twinning in the  $\alpha$  phase which act on the  $\beta \rightarrow \alpha'$  transformation systems are only present in a two-phase alloy. In the single-phase  $\beta$  alloy these stresses are not present, therefore twinning is the predominant deformation mechanism in the single phase Ti-14.8V  $\beta$  alloy rather than stress induced martensite.

#### 3.4.3 Stress induced martensite in Ti-8.1V but not Ti-6.0Mn

The third phenomenon of interest is the formation of stress induced martensite in the  $\beta$  phase of Ti-8.1V, but not in the  $\beta$  phase of Ti-6.0Mn. The  $\alpha$  phases of these two alloys are similar and deform by similar mechanisms, but deformation in the  $\beta$  phases is drastically different. The reasons for different  $\beta$  deformation behavior include differences in the strength ratio of the  $\beta$  to  $\alpha$  phases as well as the  $\beta$  phase stability and the presence of  $\omega$  phase. The effect of these factors will be discussed in detail below.

#### 3.4.3.1 β to α phase yield strength ratio

The  $\beta$  to  $\alpha$  phase yield strength ratio can play an important role in the  $\beta$  phase deformation behavior of two-phase titanium alloys. The  $\beta$  phase deforms plastically if the total stress on  $\beta$ , including the applied stress, interaction stress, and stress fields due to deformation products in the  $\alpha$  phase is greater than its yield strength. If the strength ratio of  $\beta$  to  $\alpha$  phases is high, then the additional stress in excess of the yield strength of the  $\alpha$  phase, in particular the contribution of stress from interaction with  $\alpha$ , must be greater in order to plastically deform the  $\beta$  phase.

In Ti-6.0Mn the ratio of the  $\beta$  to  $\alpha$  values of YS is ~4, whereas the Ti-8.1V has  $\beta$  to  $\alpha$  YS ratio of ~2.65. The tensile stresses applied to each two-phase alloy were comparable (623 MPa vs. 597 MPa), and the  $\alpha$  phases of each alloy deform by the same mechanisms, resulting in similar stresses from interaction and  $\alpha$  deformation products. The result of a lower  $\beta$  to  $\alpha$  stress ratio in Ti-8.1V is that the additional stress required to deform  $\beta$  plastically, once  $\alpha$  has begun to deform plastically, is much less than the increase in stress required for Ti-6.0Mn. While this required stress level is achieved for the  $\beta$  phase of Ti-8.1V, and stress induced martensite is the result, the stress level on the  $\beta$  phase of Ti-6.0Mn is insufficient for the formation of stress induced martensite. Strain from  $\alpha$  phase deformation is instead relieved in Ti-6.0Mn by interphase interface sliding.

#### **3.4.3.2** β Phase Stability and ω Phase

The martensitic transformation occurs at a temperature  $M_s$ , at or below which the transformation will happen spontaneously. Stress induced martensite occurs because the application of stress to the material effectively raises the transformation temperature  $M_s$  to a temperature equivalent to the test temperature [30-32, 79]. The change in Gibbs free energy associated with the formation of martensite,  $\Delta G$ , can be expressed as [79]:

$$\Delta G = A\gamma + V\Delta G_s - V\Delta G_v \tag{3.3}$$

where *A* is the surface area of the martensite,  $\gamma$  is the interfacial free energy,  $\Delta G_s$  is the strain energy, *V* is the volume of the nucleus, and  $\Delta G_v$  is the volume free energy release. In comparing  $\beta$  titanium alloys with different stabilities, the less stable alloys will have a greater change in volume free energy upon transformation. Therefore martensitic transformation is more likely in lower stability  $\beta$  alloys. As stated previously, the MoE has been calculated for the  $\beta$  single phases of Ti-6.0Mn and Ti-8.1V, as 19.9 for Ti-13.0Mn and 9.9 for Ti-14.8V [61]. The  $\beta$  phase of the Ti-6.0Mn is more stable than that of the Ti-8.1V alloy, so the transformation to martensite is more likely in the  $\beta$  phase of Ti-8.1V.

The presence of athermal  $\omega$  phase is a result of the instability of the  $\beta$  phase. Athermal  $\omega$  phase is present as nanostructured crystals within the  $\beta$  phase of Ti-8.1V whereas only faint traces of  $\omega$  phase are present in the Ti-6.0Mn alloy. A comparison of the selected area diffraction patterns in Figure 3.18 shows that the  $\omega$  reflections are sharp in the Ti-8.1V SADP and diffuse in the Ti-6.0Mn SADP. Streaking in the Ti-6.0Mn pattern is known as pre-omega phase streaking, and is the result of incompletely formed, or incommensurate,  $\omega$  phase [1, 13].

Kuan et al. [17] suggest that the metastable  $\omega$  phase is an intermediate phase between the  $\beta$  and  $\alpha$  phases. It is therefore likely that when  $\omega$  phase is present the activation energy as well as the free energy change required to form martensite in the  $\beta$ phase is lowered. The more stable  $\beta$  phase of Ti-6.0Mn requires higher stresses in order to overcome the higher activation energy to form stress induced martensite. Because this stress condition is not met, stress induced martensite does not form, and instead interphase interface sliding relieves the strain from slip and twinning in the  $\alpha$  phase in this alloy.





In order to assess the relationship of  $\beta$  phase stability to the type of deformation product, the tensile deformation products of Ti-V alloys with varying amounts of vanadium were reviewed. Several types of deformation products have been reported in single phase Ti-V alloys with vanadium contents ranging from 5-40wt%, including slip, twinning, several types of stress induced martensite, and stress induced  $\omega$  phase plates. Ramesh et. al [25] found (332)(113) type twins containing two variants of the  $\omega$  phase in single phase Ti-14.8wt%V alloy, with the same chemistry as the  $\beta$  component of the Ti-8.1wt% alloy. This result is consistent with the results of Oka and Teniguchi [23] in a Ti-15.5V alloy. Koul and Breedis [32] found that the stress induced plates in Ti-16.2V were hexagonal martensite,  $\alpha'$ . Menon and Krishnan [6] investigated Ti-V alloys with 5, 10 and 20wt%V, and found stress induced martensite in the Ti-20wt%V alloy. Kuan et al. [17] reported that stress induced plates in single crystal Ti-15at %V and Ti-19at%V alloys were composed of one variant of the  $\omega$  phase, with just small amounts of  $\alpha'$  present within the plate. A large number of  $\omega$  phase plates with just small amounts of  $\alpha'$  present within the plate were found in Ti-19V, but Ti-15V deformed almost entirely by slip. It should be noted that slip was the predominant deformation mechanism in one orientation of load, whereas the formation of  $\alpha'$  was the mechanism in another, indicating the importance of grain orientation with respect to the applied stress. Ling et al. [5] investigated Ti-V alloys ranging from 20 to 40 wt% V and found that as the solute concentration was increased the deformation mechanisms changed from twinning in the 20%V alloy to fine slip and finally coarse slip in the Ti-40V alloy. It appears that the deformation products are related to the amount of  $\beta$  stabilizing element, and hence the stability, in the  $\beta$  phase. As the vanadium concentration, and therefore stability, is increased, the predominant deformation mechanisms change from stress induced martensite to  $\omega$  phase plate formation, to twinning, and finally slip. This is most likely due to the relationship between the martensite start temperature,  $M_s$ , and the amount of stabilizing element. As the amount of  $\beta$  stabilizing element is increased, the M<sub>s</sub>

temperature decreases, which makes the formation of stress induced martensite more difficult. At higher stabilities the stress to deform by twinning and slip is lower than that to form stress induced martensite. The  $M_s$  temperature of the  $\beta$  phase of Ti-8.1V is higher than the  $M_s$  temperature of the  $\beta$  phase of Ti-6.0Mn [63], therefore the formation of stress induced martensite is favored in Ti-8.1V.

#### **3.5. Summary – deformation sequence**

It is likely that the following sequence of events in the material results in the series of martensite plates in the  $\beta$  phase and coarse slip or twins in the  $\alpha$  phase of Ti-8.1V. Stress in the weaker  $\alpha$  phase is increased over the yield stress by the combination of applied stress and elastic interaction stresses. This stress initiates slip or twinning within the softer  $\alpha$  phase. The combined stresses of the applied stress, interaction stress, and either dislocation pileup or twinning in the  $\alpha$  phase, along with  $\alpha$  phase templating, is sufficient to initiate the formation of martensite plates in the  $\beta$  phase of Ti-8.1V. This stress is insufficient to cause stress induced martensite in the  $\beta$  phase of Ti-6.0Mn due to its higher stability and higher  $\beta$  to  $\alpha$  strength ratio.

Two possible deformation sequences may occur. In one scenario, plastic deformation may initially occur in the  $\alpha$  phase of multiple, similarly oriented  $\alpha$  grains. Stress induced martensite then forms in the  $\beta$  phase that is between these  $\alpha$  grains after sufficient stress is accumulated from slip or twin growth in the  $\alpha$  phase. Slip and twinning were observed in the absence of martensite in Ti-6.0Mn, so this scenario seems plausible. Another possibility is that slip or twinning occurs in a single  $\alpha$  grain, which initiates martensite in the neighboring  $\beta$  grain, which in turn initiates slip or twinning in the

following  $\alpha$  grain. In this way the sequence of martensite-twin-martensite or martensiteslip-martensite "leapfrogs" from  $\beta$  to  $\alpha$  to  $\beta$  and so forth. The evidence for this possibility includes the much larger number of twins observed in the  $\alpha$  phase of Ti-8.1V compared to those in Ti-6.0Mn. The presence of stress induced martensite in Ti-8.1V causes more extensive twinning in the  $\alpha$  phase, due to the alignment of the martensite and twin shear systems, as discussed above. In either case martensite is observed to coincide with slip or twins in the  $\alpha$  phase over a distance of multiple  $\beta$  and  $\alpha$  grains.

Due to the Burgers orientation relationship of the Widmanstätten  $\alpha$ - $\beta$  alloy, it is common to have multiple  $\alpha$  platelets oriented similarly within a prior  $\beta$  grain [72]. The resolved shear stress for multiple nearby  $\alpha$  platelets is then likely to be favorable for slip or twinning, but predominantly one or the other in a given area. Therefore, slip or twinning can be expected to occur simultaneously in a number of similarly oriented  $\alpha$ grains. This trend was observed during the course of the TEM investigation. Regardless of whether slip twinning is the local  $\alpha$  deformation product, stress induced martensite forms in the  $\beta$  phase at a given critical stress depending on the strength and stability of the  $\beta$  phase.

#### **3.6.** Conclusions

These results are applicable to any two-phase alloy with a Burgers orientation relationship between phases. Unexpected deformation mechanisms may be the result of elastic interaction effects, deformation products in an adjacent phase, differences in the strength ratio between phases, and the stability of either phase. The specific conclusions from this study are:

- 1. The tensile deformation mechanisms of both phases of two-phase Ti-Mn and Ti-V titanium alloys were found to differ greatly from those of the single phase alloys that constitute the  $\alpha$  and  $\beta$  phases.
- Twins were found to form in thin α platelets of both Ti-6.0Mn and Ti-8.1V. This is in contrast to single-phase α alloys, where slip was the predominant deformation mechanism for alloys with small grain sizes. This behavior is attributed to elastic interaction stresses between the α and β phases.
- Stress induced hexagonal martensite forms in the β phase of the α+β alloy Ti-8.1V. In contrast, twinning and slip are the deformation mechanisms in the single phase β alloy, Ti-14.8V. This has been attributed to elasto-plastic interaction stresses, the α phase acting as a template for α', and the resolved shear stresses from slip and twinning in the α phase acting on the β→ α' shear systems.
- 4. Tensile deformation mechanisms are limited to slip and twinning in the α phase of Ti-6.0Mn, with no significant deformation products in the β phase, whereas the deformation products of Ti-8.1V included slip and twinning in the α phase, and stress induced hexagonal martensite (α') in the β phase. This is attributed to differences in β to α strength ratio and stability of the β phases.
- 5. The alternating sequence of slip or twins in the α phase and stress induced martensite in the β phase of Ti-8.1V may occur by slip or twinning in many α grains initially, with martensite occurring after sufficient stress builds up, or by "leapfrogging" of stress induced martensite plates in β and slip or twins in α. Such a combination of twins in the α phase and stressed induced martensite in the β phase has not been reported previously.

#### Chapter 4

## Influence of the Second Phase on the Room Temperature Deformation Mechanisms of α-β Titanium Alloys with Widmanstätten Microstructure

#### **Part 2: Creep Deformation**

In Part 1 (Chapter 3) it was observed that the presence of a second phase can affect the tensile deformation properties of titanium alloys. The present study focuses of the effect of the second phase on the ambient temperature creep deformation mechanisms of titanium alloys. A detailed investigation of the ambient temperature creep deformation mechanisms of  $\alpha$ - $\beta$  titanium alloys was performed using Ti-6.0wt%Mn and Ti-8.1wt%V with Widmanstätten microstructures as the model two-phase systems. The creep deformation mechanisms of the two-phase alloys differ from the mechanisms in single-phase alloys with compositions matching those of  $\alpha$  and  $\beta$  component phases in the two-phase alloys. These  $\alpha$ - $\beta$  deformation mechanisms include twinning in fine grains of the  $\alpha$  phase and stress induced hexagonal martensite in the  $\beta$  phase of Ti-8.1V. This is the first time that stress induced martensite is reported as a creep deformation mechanism in an  $\alpha$ - $\beta$  titanium alloy. The effect of the  $\alpha$  phase on the deformation mechanisms of the  $\beta$ phase, and vice-versa, has been investigated. Several factors contribute to the creep deformation mechanisms in the model two-phase alloys, including elastic interaction effects, shear stress due to deformation products in adjacent phases, and the stability of the  $\beta$  phase. Models for the time dependent growth of martensite are suggested.

#### 4.1. Introduction

In many applications creep resistance at low temperatures ( $<0.25T_m$ ) is an important property. For example, a recent study performed by the Nuclear Regulatory Commission showed titanium alloys in a drip shield proposed for use in the Yucca Mountain nuclear waste repository may sustain loads approaching their yield stress at ambient or slightly elevated temperatures due to rock fall, and would be at risk of failing by creep deformation [81].

Titanium and titanium alloys have been shown to creep at stress levels as low as 25-60% of their 0.2% yield stress (YS) at ambient temperature [43, 45, 54, 55]. This was first observed in commercial purity titanium, but was later observed in alloys. Although early studies were primarily phenomenological [55, 56, 82], more recently the low temperature creep deformation mechanisms have been investigated for single and two-phase titanium alloys. Creep in the single phase  $\alpha$  and  $\beta$  alloys has been attributed to slip [46, 60, 83-85] as well as twinning [57, 58, 60, 85]. The deformation mechanisms of the  $\alpha$ - $\beta$  alloys has been attributed to slip in the  $\alpha$  and  $\beta$  phases as well as across the  $\alpha$ - $\beta$  interfaces [42-48]. Interphase interface sliding has also been reported [45, 49, 50].

In contrast to the tensile plastic deformation of titanium alloys, creep deformation may take place at stresses below the yield stress of the material, where only a finite level of applied stress is available for the activation of creep deformation mechanisms. Any stresses in excess of the applied stress which may contribute to the activation of creep deformation processes are only available from internal sources, such as interactions between phases. These interactions are the focus of the current work. Interactions between phases result in additional deformation mechanisms in both the  $\alpha$  and  $\beta$  phases,

leading to increased creep strain. In order to design alloys and processes for improved creep resistance, these interactions must be understood.

#### **4.2.** Experimental procedure

Ti-Mn and Ti-V alloys were selected and prepared as described in Chapter 2. Ambient temperature (298K) creep tests were performed on an ATS lever arm creep testing machine at a constant load equal to 95% and 100% of each alloy's respective YS, which was determined during the ambient temperature tensile tests described in Chapter 3. A clip on extensometer was used to record strain. Optical and SEM micrographs were taken prior to and following testing in the same area of each specimen to record deformation mechanisms.

Following creep testing, TEM specimens were prepared from the gage length (deformed material) and from the grip section (undeformed material) of specimens tested at 95% YS using the procedure given in Chapter 2.7. The undeformed material was examined to ensure that the observed deformation mechanisms were a result of testing and not artifacts of TEM specimen preparation. A JEOL JEM-4000FX transmission electron microscope, operating at 300 KeV, was used to obtain the images and diffraction patterns for this study.

#### 4.3. Results

#### 4.3.1 Single-phase α and β alloys

The single-phase  $\alpha$  and  $\beta$  alloys have been creep tested at 95% of their respective yield stresses in several previous studies. The creep strain of the single-phase alloys depended on grain size and stability of the  $\beta$  phase alloys [60, 61]. All of the single-phase alloys showed creep exhaustion behavior during the test periods.

The single-phase  $\alpha$  alloy Ti-0.4Mn ( $\alpha$  phase of Ti-6.0Mn) with large grain size (500µm) crept to a strain of 2.7%, and deformed by slip, instantaneous twinning and time dependent twinning [27, 57, 59]. Time dependent twinning was also observed in the same alloy crept in compression [58]. The same alloy with a small grain size (45µm) crept to a strain of 0.65%, and deformed primarily by slip, with very few twins and infrequent grain boundary sliding [27]. The single-phase  $\beta$  alloy Ti-13.0Mn ( $\beta$  phase of Ti-6.0Mn) with a grain size of 200µm showed negligible creep strain of only .03% strain over a test period of 400 hours, and deformed only by rare coarse slip, with no observed twinning [57, 59].

The single-phase  $\alpha$  alloy Ti-1.6V ( $\alpha$  phase of Ti-8.1V) with a grain size of 226µm crept to a strain of 6.67% and deformed by fine slip, instantaneous twinning and timedependent twinning [85]. The single-phase  $\beta$  alloy Ti-14.8V ( $\beta$  phase of Ti-8.1V) with a large grain size (350µm) crept to a strain of 0.101% and deformed by slip, instantaneous twinning and time dependent twinning. Small grain Ti-14.8V alloys (18-25µm) crept to 0.02% strain and deformed primarily by slip, with no significant twinning [28, 60].

#### **4.3.2** Two-phase α-β alloys

Figure 4.1 shows the creep curves of Ti-6.0wt%Mn and Ti-8.1wt%V  $\alpha$ - $\beta$  alloys creep tested at 95% and 100% of their respective 0.2% YS [27, 75]. The Ti-8.1wt%V alloy exhibits a greater creep strain than does the Ti-6.0wt%Mn alloy at both stress levels. This difference in creep strain is significant even at the 95% stress level. Ti-8.1V has 16% greater creep strain compared to Ti-6.0Mn when the instantaneous plastic deformation of ~0.23% is subtracted from the total strain of each alloy. The difference in strain is even higher at 32% with alloys tested at 100%YS. This significant difference in creep strain is attributed to a difference in creep deformation mechanisms. The following analysis of creep deformation mechanisms was performed on specimens crept at 95% YS.



**Figure 4.1.** Creep curves of Ti-6.0wt%Mn and Ti-8.1wt%V alloys, creep tested at ambient temperature at 95% and 100% of their respective YS [27, 75].

#### 4.3.2.1 Creep deformation mechanisms

Creep deformation of Ti-6.0Mn is primarily due to fine slip in the  $\alpha$  phase, which is evidenced by a widening of the gold fiducial grid, as measured from before and after SEM micrographs, and TEM analysis. Infrequent interphase interface sliding was observed by SEM [27]. No twinning or stress-induced martensite was observed in either phase by TEM, although it is possible that some micro-twins are present in the  $\alpha$  phase in a region of specimen not investigated. This notion seems likely, as twins were present in the  $\alpha$  phase of the same alloy deformed in tension, but the strain of the creep tested specimen is much less. The results of the previous studies of the single-phase  $\alpha$  and  $\beta$ alloys indicate that the tensile and creep deformation mechanisms are typically similar, only less frequent for creep deformed specimens due to the lower total strain. Figure 4.2 shows SEM micrographs taken of the surface of Ti-6.0Mn prior to and following creep testing at 95% YS. TEM investigations showed extensive 'a' type slip on the prism planes of the  $\alpha$  phase, which is shown in Figure 4.3. No slip, twinning or stress-induced martensite (SIM) was observed in the  $\beta$  phase of this alloy, which is not surprising considering the limited creep deformation of the single-phase  $\beta$  alloy Ti-13.0Mn.



**Figure 4.2.** SEM micrographs of  $\alpha$ - $\beta$  Ti-6.0Mn (a) before and (b) after creep deformation at 95%YS for 280 hours to 0.48% plastic strain at 95% YS. There are no coarse deformation features visible on the polished and etched surface, although fine slip was noted due to an increase of the fiducial line spacing during testing [27].



**Figure 4.3.** TEM Dark field micrograph of fine slip lines in the  $\alpha$  phase of Ti-6.0Mn. Slip is 'a' type dislocations on prism planes. Dislocations with three different Burgers vectors of the type  $\vec{b} = 1/3\langle 11\overline{2}0 \rangle$  are slipping simultaneously.

In contrast to the creep deformation mechanisms of Ti-6.0Mn, coarse deformation features were present in creep tested samples of Ti-8.1V. Figure 4.4 shows SEM micrographs taken prior to and following creep deformation of Ti-8.1V at 95% YS. Notice the coarse deformation products that traverse the  $\alpha$  and  $\beta$  phases over a distance of many grains. Previously, these deformation features were incorrectly identified as coarse slip bands crossing the  $\alpha$ - $\beta$  interfaces on parallel slip systems [27, 75]. Further investigation using TEM analysis during the course of the present investigation revealed that these coarse deformation features are actually twins or coarse slip in the  $\alpha$  phase, and stress-induced hexagonal martensite,  $\alpha'$ , in the  $\beta$  phase.

This is the first time that stress induced martensite has been reported in a creep deformed two-phase titanium alloy at ambient temperature. Further, this stress induced martensite was frequently observed in combination with twins in the  $\alpha$  phase, which has

not been reported in any creep tested two-phase titanium alloy. These deformation mechanisms are responsible for the greater creep strain of Ti-8.1V compared to Ti-6.0Mn.

Figure 4.5 shows an example of the coarse slip that was present in the  $\alpha$  phase, and Figure 4.6 is a dark field image identifying similar coarse slip in another area as 'a' type prism slip. Figure 4.7 shows several twins in the  $\alpha$  phase in combination with a stress induced martensite plate in the  $\beta$  phase, which touches the  $\alpha$  interface. The twin labeled T<sub>1</sub> was identified as a  $\{10\overline{1}2\}\langle\overline{1}011\rangle$  type twin. The other twins were twinned in a different plane, and thus identification was not possible in this grain. Figure 4.8 shows two parallel twin-martensite pairs traversing the width of  $\alpha$  and  $\beta$  grains. Figure 4.9 shows martensite across the width of a  $\beta$  grain with twins growing primarily down the length of the  $\alpha$  grain. The accompanying diffraction patterns identify the hexagonal stress-induced martensite, and show a  $\{10\overline{1}1\}$  twin relationship between the martensite and the adjacent  $\alpha$  phase, which will be discussed further. Figure 4.10 shows another example of stress induced martensite in  $\beta$  in combination with a twin in  $\alpha$ . These martensite plates spanned the length of the  $\beta$  phase parallel to the  $\alpha$  plates, demonstrating that the martensite can form in various orientations with respect to the grains, which is dependent on the grain orientation to the loading axis.

These results are unexpected when compared to the creep deformation behavior of the single-phase alloys for several reasons. First, the grain size of both the  $\alpha$  and  $\beta$ phases are relatively small, and any significant stress induced plate formation, twinning or martensite, is unexpected at small grain sizes due to a large nucleation stress [26, 30]. This is more remarkable when one considers that the applied stresses are only 95% the

yield stress. Second, in a previous study of the creep deformation behavior of Ti-14.8V, the  $\beta$  phase of Ti-8.1V, the primary deformation mechanism was {332} twinning [60]. No stress-induced martensite was observed. This difference in deformation mechanisms suggests that the presence of the  $\alpha$  phase alters the creep deformation mechanisms of the  $\alpha$  and  $\beta$  phases in a two-phase titanium alloy.







**Figure 4.5.** TEM bright field micrograph of slip bands in the  $\alpha$  phase of  $\alpha$ - $\beta$  Ti-8.1V.



**Figure 4.6.** TEM dark field micrograph of slip band in the  $\alpha$  phase of Ti-8.1V. Slip occurs with 'a' type screw dislocations on prism planes.



**Figure 4.7.** Bright field TEM micrograph of Ti-8.1V showing several twins in the  $\alpha$  phase along with a stress induced martensite plate in the  $\beta$  phase. Accompanying diffraction patterns from (a) the  $\alpha$  phase (b) the twin T<sub>1</sub> (c) the T<sub>1</sub>/ $\alpha$  interface. T<sub>1</sub> is a  $\{10\overline{1}2\}$  type twin. Selected area diffraction from (d) the  $\beta$  phase shows the Burgers OR with the  $\alpha$  phase. The (e) hexagonal stress induced martensite plate  $\alpha'$  has a  $(10\overline{1}\overline{1})$  twin relationship to the  $\alpha$  phase, illustrated by the (f)  $\alpha/\alpha'$  interface pattern. Zone axis is  $[1\overline{2}10]_{\alpha} //[1\overline{1}1]_{\beta}$ .



**Figure 4.8.** Bright field TEM micrograph showing stress induced martensite plates in the  $\beta$  phase and twins in the  $\alpha$  phase of Ti-8.1V. The martensite plates and twins alternate across the width of the  $\beta$  and  $\alpha$  grains.



**Figure 4.9.** Bright field TEM micrograph of stress induced hexagonal martensite ( $\alpha'$ ) plates in the  $\beta$  phase and twins in the  $\alpha$  phase of Ti-8.1V, indicated by arrows. To the right are selected area diffraction patterns taken from the (a)  $\beta$  phase (b)  $\alpha'$  plate (c) across the  $\alpha'/\beta$  interface and (d)  $\alpha$  phase. The  $\alpha'$  plate and the  $\alpha$  phase have a  $\{10\overline{1}1\}$  twin relationship. Zone axis is  $[1\overline{2}10]_{\alpha} //[1\overline{1}1]_{\beta}$ .



Figure 4.10. Bright field TEM micrograph showing two stress-induced martensite plates in the  $\beta$  phase contacting a twin in the  $\alpha$  phase.

#### 4.4. Discussion

#### 4.4.1 Contribution of deformation mechanisms to creep strain

The creep strain of Ti-8.1V was greater than that of Ti-6.0Mn over the same test period. This additional creep strain can be explained by a difference in deformation mechanisms, namely the formation of stress-induced martensite in the  $\beta$  phase and a greater number of twins in the  $\alpha$  phase of Ti-8.1V.  $\{10\overline{1}2\}$  type twins were identified, but it is also likely that  $\{10\overline{1}1\}$  twins are also present, as they were present in the same alloy deformed in tension, and past studies of the single phase alloys show that the tensile and creep deformation products are similar, albeit in higher concentrations in tensile specimens due to their greater plastic strain. The  $\alpha$  phases of both alloys deformed by extensive fine slip, with some coarse slip also observed in the  $\alpha$  phase of Ti-8.1V. No

significant slip was observed in the  $\beta$  phase of either alloy. An understanding of the creep deformation mechanisms in two-phase titanium alloys due to  $\alpha$ - $\beta$  interactions is essential in order to design and process alloys for reduced creep strain.

#### 4.4.2 Effect of β phase stability on the formation of stress induced martensite

As the stability of the  $\beta$  phase is reduced, stress induced martensite is more likely to form due to an increase in the M<sub>s</sub> temperature [30]. Higher stability  $\beta$  alloys deform by twinning and slip.

The molybdenum equivalency (MoE) is a relative comparison of the stability of a  $\beta$  alloy as compared to a Ti-Mo alloy, expressed in terms of wt% Mo in a Ti-Mo alloy with the same stability. Molybdenum is a  $\beta$  phase stabilizer, thus a higher MoE equates to a more stable  $\beta$  phase. In comparing the stability of the  $\beta$  phases of Ti-6.0Mn and Ti-8.1V, the MoE has been calculated as 9.9 for the  $\beta$  phase of Ti-8.1V and 19.9 for the  $\beta$  phase of Ti-6.0Mn [61].

The lower  $\beta$  phase stability of Ti-8.1V is partially responsible for the formation of SIM during creep deformation, but is not the only factor, as the single-phase  $\beta$  Ti-14.8V alloy deformed by twinning. Stress induced martensite was the creep deformation mechanism for Ti-14.8V only in the presence of the  $\alpha$  phase, the effects of which are discussed in Section 4.3. However, the higher stability of the  $\beta$  phase of Ti-6.0Mn precludes the formation of SIM, even in the presence of the  $\alpha$  phase.

In  $\beta$  alloys with low stability, athermal  $\omega$  phase forms upon quenching. This transformation cannot be suppressed regardless of the cooling rate. The  $\omega$  phase is considered to be an intermediate phase in the  $\beta$  to hexagonal martensite transformation

[17], therefore the presence of  $\omega$  phase may contribute to martensite formation in Ti-8.1V.

# 4.4.3 Differences in the single and two-phase creep deformation mechanisms – effect of the second phase

Creep deformation mechanisms in two-phase alloys differ from those observed in the component single-phase alloys. First, significant twinning was not observed in singlephase alloys with fine (<45 $\mu$ m) grains. Twinning occurs in the  $\alpha$  phase of both two-phase alloys, more so in Ti-8.1V, which have average grain widths of less than 10µm, with many grains having widths less than 1 $\mu$ m. Second, in the single-phase  $\beta$  alloy Ti-14.8V, which has the same composition as the  $\beta$  phase of the two-phase alloy Ti-8.1V (see Table 4.I), the stress-induced plates responsible for creep deformation were identified as {332} type twins [60]. These twins contained both orientations of the  $\omega$  phase seen within the parent crystal. In contrast, within the  $\beta$  phase of the two-phase Ti-8.1V alloy, hexagonal stress-induced martensite plates  $(\alpha')$  formed with the Burgers orientation relationship to the  $\beta$  phase of  $\langle 1\overline{2}10 \rangle (0001)_{\alpha'} // \langle 1\overline{1}1 \rangle (110)_{\beta}$ . No  $\omega$  phase was observed within the martensite plates, therefore the  $\omega$  phase was completely transformed along with the  $\beta$ phase during the martensitic transformation. No twinning was observed in the  $\beta$  phase of the  $\alpha$ - $\beta$  Ti-8.1V. As a single phase, Ti-14.8V has a martensite start (M<sub>s</sub>) temperature sufficiently low to prevent a stress induced martensitic transformation. The presence of the  $\alpha$  phase in effect raises the M<sub>s</sub> temperature, such that martensite can form during creep deformation.

Both the  $\alpha$  and  $\beta$  phase creep deformation mechanisms were altered due to the presence of a second phase, which affects the creep strain. Various contributing factors for the observed creep deformation mechanisms are presented below.

#### 4.4.4 The special importance of interaction stresses during creep deformation

Interaction stresses between the  $\alpha$  and  $\beta$  phases are responsible for the deformation mechanisms of the two-phase alloys, which differ from those of the singlephase alloys that constitute the individual phases. This difference in deformation mechanisms was first noted for specimens deformed in tension, as discussed in Chapter 3. The stress applied to a specimen tested in tension is continually increased in order to achieve a desired strain rate. In contrast, specimens examined in this study were creep tested at a constant 95% of their respective YS values. Any additional stresses required to initiate creep processes must be from internal sources. Therefore, internal stresses due to interactions between the  $\alpha$  and  $\beta$  phases are even more important during creep deformation at low stresses compared to tensile deformation. There are several interaction stresses between the  $\alpha$  and  $\beta$  phases that must be considered. These interactions are discussed, along with their consequences, below.

#### 4.4.4.1 Elastic interaction stresses

As the creep specimens are loaded to 95% of their respective yield stresses, there is an instantaneous elastic strain. The effect of elastic interactions on  $\alpha$ - $\beta$  alloys with a Widmanstätten microstructure deformed in creep is then similar to alloys deformed in tension as discussed in Chapter 3, Section 4.1.1 and 4.2.1. These stresses initially

increase stress in the  $\alpha$  phase, contributing to the formation of twins in narrow grains, even at the relatively low applied creep stress. After significant  $\alpha$  phase deformation, elasto-plastic interaction stresses can act on the  $\beta$  phase, contributing to stress induced martensite nucleation.

#### 4.4.4.2 α phase as a template for stress induced hexagonal martensite

The presence of the  $\alpha$  phase is responsible for the formation of hexagonal SIM instead of twins in the  $\beta$  phase of Ti-8.1V. TEM analysis revealed a  $\{10\overline{1}1\}$  twin relationship between martensite plates in the  $\beta$  phase and the adjacent  $\alpha$  phase of creep deformed specimens. The selected area diffraction patterns taken from the martensite plate and the  $\alpha$  phase in Figure 4.7 a, e and f and 4.9 b and d demonstrate this relationship. The identical crystal structure and the observed twin relationship between the  $\alpha$  and  $\alpha'$  makes the  $\alpha$  phase an exceptional template for the nucleation of stress induced hexagonal martensite in the  $\beta$  phase in alloys with a Widmanstätten microstructure. There is no such orientation relationship or crystallographic similarity between the  $\alpha$  phase and  $\{332\}$  twins in the  $\beta$  phase. Therefore the interface created between the a phase and the stress induced martensite plate is expected to be coherent, whereas the interface between  $\{332\}$  twins in  $\beta$  and the  $\alpha$  phase would not be. SIM formation would be favored over twinning in  $\beta$  due to a lower misfit strain and decreased surface free energy for martensite formation.

#### 4.4.4.3 Alignment of α phase twin and β phase martensite shear systems

Deformation products in the  $\alpha$  phase, slip and twinning, act on various shear systems in the  $\beta$  phase due to the Burgers orientation relationship between the  $\alpha$  and  $\beta$ phases. It is known that during creep deformation of two-phase titanium alloys slip in the  $\alpha$  phase can initiate slip in the  $\beta$  phase [42-48]. In the case of Ti-8.1V, stress from deformation products in the  $\alpha$  phase initiates stress induced martensite in the  $\beta$  phase during creep deformation. Calculations which were performed as part of the tensile deformation study in Chapter 3 are applicable to the observed creep deformation because of the similar mechanisms of  $\alpha$  and  $\beta$  phase deformation. The complete results of these calculations are available in Appendix J. It was found that the magnitude of resolved shear stresses from multiple slip and twin shear systems in the  $\alpha$  phase was greater on the  $\beta$  phase  $\beta \rightarrow \alpha'$  shear systems than the  $\beta$  twin shear systems. In this regard, the presence of the  $\alpha$  phase results in SIM rather than twinning in the  $\beta$  phase.

In turn, stress from SIM in the  $\beta$  phase acts upon slip and twin shear systems in the  $\alpha$  phase. Twinning in  $\alpha$  was most common in creep deformed Ti-8.1V when accompanied by martensite in an adjacent  $\beta$  grain as part of an extended sequence of martensite-twin-martensite, although twins could be present without direct interaction with SIM at the  $\alpha$ - $\beta$  interface, as shown in Figure 4.11. This suggests that while the combination of applied and elastic interaction stress is sufficient only to nucleate limited  $\alpha$  phase twinning (as evidenced by Ti-6.0Mn), SIM in the  $\beta$  phase acting on the twinning shear system in the  $\alpha$  phase generates many more twins during creep deformation.



**Figure 4.11**. Bright field TEM micrograph showing a stress induced martensite plate (indicated by black arrow) in the  $\beta$  phase along parallel to the  $\alpha$  plate length, with a twin (indicated by white arrow) in the adjacent  $\alpha$  phase of Ti-8.1V.

#### 4.4.4.4 Effect of the β phase on limiting α phase twinning

In order for extensive twin nucleation and growth in the  $\alpha$  phase, the  $\beta$  phase must strain to accommodate  $\alpha$  phase strain. If the  $\beta$  phase constrains the  $\alpha$  phase sufficiently, the formation and growth of twins will be retarded. This is evidenced in the current study. Widespread twinning occurs in the  $\alpha$  phase of Ti-8.1V, but rarely in Ti-6.0Mn. The  $\beta$ phase of Ti-8.1V deforms by stress-induced martensite, which relieves strain in  $\alpha$ , and twinning can proceed. In Ti-6.0Mn, the high strength and high stability of the  $\beta$  phase prevent the formation of stress-induced martensite. The limited interphase interface sliding of Ti-6.0Mn is insufficient to completely relieve the  $\alpha$  phase stress, which prevents the growth of twins and limits the overall creep strain, although some twinning could occur due to elastic strain in  $\beta$ . In specimens deformed in tension strain and the amount of sliding is greater, therefore more twinning was observed in the  $\alpha$  phase of tensile deformed specimens.

#### 4.4.5 Time-dependent vs. instantaneous deformation mechanisms

The time dependency of creep deformation mechanisms contrasts the "instantaneous" tensile deformation mechanisms. Several time-dependent mechanisms have been observed during room temperature creep of titanium alloys, including slip [42-48, 60, 83-85], interphase interface sliding [45, 49, 50], and time-dependent twinning. Time dependent twinning has been observed in single-phase  $\alpha$  and  $\beta$  alloys [57, 58, 60, 85]. The slow growth of time dependent twins has been attributed to the diffusion of interstitial oxygen away from the advancing twin front from interstitial sites that are not conserved in the twin, limiting the growth rate [86]. It is likely that twins in the  $\alpha$  phase of the two-phase alloys in the present study are also time-dependent, as time dependent twins were observed in their single-phase  $\alpha$  counterparts.

The extensive stress-induced martensite observed in the  $\beta$  phase of Ti-8.1V contributes to the creep strain, but there is some question as to whether this martensite grows instantaneously or is also time-dependent. The first and most likely possibility is that the martensite grows instantaneously after sufficient stress contributions from time-dependent creep processes, either slip or twinning, in the  $\alpha$  phase.

The second possibility is that the rate of martensite plate growth is controlled by the growth of twins in the adjacent  $\alpha$  phase. Because the growth of twin width is due to sequential shearing of adjacent atomic planes in  $\alpha$  [87, 88], the stress on the  $\beta$  phase increases in proportion to the twin width. Martensite would nucleate at some critical

stress due to a twin in  $\alpha$  reaching a critical width. At this point the martensite would no longer grow without additional stress from the twin in the  $\alpha$  phase. As the twin continues to grow slowly with time, a shearing of twin planes in  $\alpha$  would result in shearing of the planes responsible for the martensite transformation in the  $\beta$  phase, thereby controlling the martensite growth rate. The very similar plate widths of twins and martensite shown in Figures 4.8, 4.9 and 4.10 suggest that this is a possible mechanism. A model of this process using  $\{10\overline{1}1\}$  twins is presented in Figure 4.12.

A similar process may occur in response to slip in the  $\alpha$  phase. The stress placed on the  $\beta$  phase is proportional to the number of dislocations that slip in  $\alpha$ . Slip in the  $\alpha$ phase would proceed until a critical number of dislocations have passed along slip planes oriented favorably with  $\beta \rightarrow \alpha'$  transformation shear systems in the  $\beta$  phase, at which time martensite would nucleate. As further slip occurs in the  $\alpha$  phase, the  $\beta$  phase will experience increasing stress proportional to the number of dislocations that glide on slip planes aligned with the martensite shear planes. With subsequent dislocation motion the martensite would grow, relieving strain in the  $\beta$  phase to accommodate slip in the  $\alpha$ phase. A model of this process is illustrated in Figure 4.13 for prism slip in the  $\alpha$  phase.



**Figure 4.12.** Schematic of martensite nucleation and growth controlled by  $\{10\overline{1}1\}$  twin growth in the  $\alpha$  phase. The  $\alpha$  phase (white) is projected onto  $(2\overline{1}\overline{1}0)_{\alpha}$ .  $\beta$  and martensite planes are out of this plane. (a) Twin has nucleated in the  $\alpha$  phase, but the stress is too low for martensite to nucleate in the  $\beta$  phase (blue). (b) Twin has grown and a critical stressed is reached for martensite (green) nucleation in the  $\beta$  phase. (c) and (d) As the twin continues to grow, shear from subsequently twinning planes in the  $\alpha$  phase causes the martensite to grow proportionally in the  $\beta$  phase.



**Figure 4.13.** Schematic of martensite nucleation and growth controlled by slip in the  $\alpha$  phase (white). Slip of 'a' type screw dislocations on the  $(01\overline{1}0)_{\alpha}$  plane is represented.  $\beta$  (blue) and martensite (green) planes are out of this plane. (a) Slip is initiated in the  $\alpha$  phase, placing stress on the  $\beta$  phase, but the magnitude is insufficient for martensite nucleation. (b) Slip has proceeded and the critical stress for martensite nucleation has been reached. (c) and (d) As slip continues to grow, stress on the  $\beta$  phase from the movement of subsequent dislocations forces martensite to grow in proportion to the number of dislocations that glide on the slip plane.

In the previous scenarios the deformation rate is controlled by the growth rate of time-dependent twins or the rate of slip. The third possibility is that the martensite could itself be time dependent, with a rate-limiting mechanism similar to time-dependent twinning. At present there is insufficient evidence to conclude that the SIM grows at any other velocity than one approaching the speed of sound in the material. This possibility warrants further study by interrupt creep tests to assess the growth rate of the SIM plates. In any of the above cases the slowest step will be the rate limiting step for creep deformation.

#### 4.5. Summary of creep deformation processes

The following sequence of events results in the creep deformation of Ti-6.0Mn and Ti-8.1V. When the specimens are first loaded to a stress equivalent to 95% of their yield stress, there is an instantaneous elastic deformation. Elastic interaction stresses are placed on the  $\alpha$  phase. The combination of the applied stress and the elastic interaction stress causes slip and/or twinning in the  $\alpha$  phase. Dislocations move slowly with time due to the relatively low applied stress, and begin to pile-up at the  $\alpha$ - $\beta$  interface. Time dependent twins can also form and grow, adding to the creep strain. As the  $\alpha$  phase deforms plastically, interaction stresses are reduced on the  $\alpha$  phase, and the  $\beta$  phase must deform or there must be interphase interface sliding in order to accommodate further  $\alpha$ phase deformation.

After sufficient time, the shear stress from the dislocation pileup or twinning is sufficient to nucleate stress-induced martensite in the  $\beta$  phase of Ti-8.1V because of aligned shear systems in the  $\alpha$  and  $\beta$  phases due to the Burgers orientation relationship.

Stress induced martensite, rather than twinning, occurs due to the  $\alpha$  phase acting as a template, and the higher resolved shear stress acting on martensite rather than twin shear systems. The stress required to nucleate stress-induced martensite is too high in Ti-6.0Mn due to its higher  $\beta$  phase stability, therefore SIM does not form in this alloy. This difference in deformation mechanisms is responsible for the lower creep strain of Ti-6.0Mn.

When the martensite spans the  $\beta$  phase, twinning in the adjacent  $\alpha$  phase can be triggered. This is once again due to aligned shear systems in the  $\alpha$  and  $\beta$  phases. This process can continue across many  $\alpha$  and  $\beta$  grains due to the Burgers orientation relationship of  $\alpha$  and  $\beta$ . Twinning in the  $\alpha$  phase may be time dependent or instantaneous. The growth rate of martensite may also be may be instantaneous, controlled by the growth rate of twins or slip rate in the  $\alpha$  phase, or the martensite may have an inherent time-dependent mechanism. Further study is warranted to explore these possibilities.

#### 4.6. Conclusions

- The creep strain of Ti-8.1wt%V was greater than that of Ti-6.0wt%Mn alloy when both alloys were tested at 95% of their respective yield stress. This is attributed to a difference in deformation mechanisms.
- Deformation mechanisms of Ti-6.0Mn are limited to fine slip and rare twinning in the α phase, and rare interphase interface sliding, whereas creep deformation of Ti-8.1V occurred by slip and twinning in the α phase, and stress induced hexagonal martensite in the β phase. This is the first time that stress induced

martensite is reported as a room temperature creep deformation mechanism in an  $\alpha$ - $\beta$  titanium alloy.

- Formation of hexagonal SIM plates during creep deformation in the β phase of Ti-8.1V only occurs in the presence of α platelets. Twinning occurs in the singlephase β alloy Ti-14.8V, which has the same chemistry as the β phase of Ti-8.1V.
- Twins in the α phase occurred in response to stress induced martensite in the β phase. This is evidenced by TEM observations of twin-martensite pairings in Ti-8.1V and the lack of twins in Ti-6.0Mn.
- Stress induced martensite contributes to the creep strain of Ti-8.1V. However, its growth may be instantaneous or time dependent. Several possible mechanisms for time dependent growth of martensite are proposed.
- 6. Interactions between phases are an extremely important consideration in creep deformation because they can be responsible for additional deformation mechanisms, resulting in increased creep strain. Understanding these interactions is important in the design and processing of two-phase alloys for improved creep resistance.

#### Chapter 5

### Studies of Interphase Interface Sliding in α-β Titanium Alloys with Widmanstätten Microstructure

 $\alpha$ - $\beta$  Ti-6.0Mn deforms by interphase interface sliding during tensile and creep testing, while no interphase interface sliding was observed in  $\alpha$ - $\beta$  Ti-8.1V. Additionally, sliding in Ti-6.0Mn and other Ti-Mn alloys only occurs on one side of an  $\alpha$  phase plate and not the other, which is termed anisotropic interface sliding. This aim of this investigation is to explore contributions to the difference in interphase interface sliding behavior of Ti-6.0Mn and Ti-8.1V, and the reason for anisotropic sliding of the  $\alpha$ - $\beta$ interfaces. The  $\alpha$ - $\beta$  interface morphology was extensively examined using TEM and HREM. A difference in the interface morphology was observed using HREM, but the effect on interface sliding behavior is inconclusive. Other contributing factors are offered. A model of mechanical locking is proposed to explain anisotropic interface sliding in  $\alpha$ - $\beta$ titanium alloys with Widmanstätten microstructure.

#### 5.1. Introduction

Interface sliding has been observed during room temperature tensile and creep deformation of Ti-6.0Mn with Widmanstätten microstructure [27]. In contrast, no interphase interface sliding was observed during room temperature tensile and creep deformation of Ti-8.1V with the same microstructure. The SEM shown in Figure 5.1 shows the surface of Ti-6.0Mn deformed in tension where interphase interface sliding is visible along the  $\alpha$ - $\beta$  interface at the areas indicated by arrows. Displacement of the
fiducial lines across the  $\alpha$ - $\beta$  interface indicates sliding. Anisotropic interphase interface sliding, sliding on only one side of the  $\alpha$  grain, has been observed in Ti-Mn alloys, including Ti-6.0Mn [27] and several Ti-Mn alloys with varying solute concentrations in a study by Ankem and Margolin [49]. This is visible in Ti-6.0Mn in Figure 5.1 as well as Ti-3.9wt%Mn following compression testing at ambient temperature to 3% strain in Figure 5.2. The Ti-6.0Mn alloy has a fine  $\alpha$  phase microstructure while Ti-3.9wt% has a comparatively coarse microstructure.

An investigation of the  $\alpha$ - $\beta$  interface structure was undertaken in an attempt to explain the difference in interface sliding behavior between Ti-6.0Mn and Ti-8.1V and anisotropic interface sliding.



**Figure 5.1.** SEM micrograph of the surface of Ti-6.0wt%Mn with a fine  $\alpha$  phase microstructure following tensile deformation. Areas indicated by arrows show where sliding has occurred, resulting in a displacement of the fiducial line across the interface. Note that sliding is only visible on one side of the  $\alpha$  phase grains [27].



**Figure 5.2.** Optical micrograph showing anisotropic interphase interface sliding in Ti-3.9wt%Mn  $\alpha$ - $\beta$  alloy with a coarse microstructure deformed in compression to 3% strain. Displacement of fiducial lines at sides marked A and C indicate sliding, where no sliding was observed at the opposite sides marked E [49].

## **5.2. Experimental Procedure**

The  $\alpha$ - $\beta$  interface structure of Ti-6.0Mn and Ti-8.1V was investigated using TEM and HREM. TEM specimens were produced from Ti-6.0Mn and Ti-8.1V alloys by the procedure given in Chapter 2.7. TEM and HREM were performed on a JEOL JEM-4000FX microscope operating at 300 KeV. For HREM images of the  $\alpha$ - $\beta$  interfaces, selected area diffraction was used to align the interface plane  $(\overline{5}140)_{\alpha}//(\overline{3}34)_{\beta}$  parallel to the electron beam direction. The interface was viewed from two perpendicular directions,  $[0001]_{\alpha}$  // $[110]_{\beta}$  and  $[1\overline{2}10]_{\alpha}$  // $[1\overline{1}1]_{\beta}$ . In the case of the  $[0001]_{\alpha}$  // $[110]_{\beta}$  the interface plane is exactly parallel the electron beam. Therefore a clear  $\alpha$ - $\beta$  interface without any overlap of phases can be viewed from this specimen orientation. For the interface viewed from  $[1\overline{2}10]_{\alpha}$  // $[1\overline{1}1]_{\beta}$  only a slight overlap of phases is expected, as this direction lies only 11.4 degrees out of the interface plane.

#### 5.3. Results and Discussion

#### **5.3.1 Investigation of α-β interface structure**

It was postulated that the relative ease of  $\alpha$ - $\beta$  interface sliding in Ti-6.0Mn compared to Ti-8.1V may be due to differences in  $\alpha$ - $\beta$  interface morphology. The  $\alpha$ - $\beta$  interfaces of Ti-8.1V and Ti-6.0Mn appeared similar when viewed by TEM at intermediate magnifications (40-150 kx), but when viewed using high resolution imaging, subtle differences in the interface roughness were visible. When viewed from the  $[000 \overline{1}]_{\alpha} //[\overline{1} \overline{1} 0]_{\beta}$  direction the interface of Ti-8.1V appears to have a slightly rough, undulating interface. A high resolution image of an  $\alpha$ - $\beta$  interface of Ti-8.1V viewed from this direction is shown in Figure 5.3. In contrast, the interface of Ti-6.0Mn appears relatively straight, which is shown in the high resolution image of the  $\alpha$ - $\beta$  interface of Ti-6.0Mn appears

Differences in the interface structure of Ti-8.1V and Ti-6.0Mn are more difficult to discern when the interface is viewed from  $[1\overline{2}10]_{\alpha} //[1\overline{1}1]_{\beta}$  due to a slight overlap of phases. While this misalignment of the viewing axis with the interface plane is only 11° and the sample is thin so the overlapping area should be extremely small, the overlap may

alter the appearance of the interface shape, which makes a conclusive observation difficult. Observations of the interfaces of Ti-8.1V and Ti-6.0Mn taken from  $[1\overline{2}10]_{\alpha}//[1\overline{1}1]_{\beta}$  seem to show a slightly rougher interface for Ti-8.1V, although both interfaces undulate slightly. High resolution images of  $\alpha$ - $\beta$  interfaces in Ti-8.1V and Ti-6.0Mn viewed from  $[1\overline{2}10]_{\alpha}//[1\overline{1}1]_{\beta}$  are presented below In Figures 5.5 and 5.6 respectively.

The contribution of interface morphology to the sliding behavior of these alloys is inconclusive. While there appears to be a slight difference in the roughness of the interface between Ti-8.1V and Ti-6.0Mn, it is unknown if this will have a significant effect on interface sliding. While a larger magnitude of surface roughness would undoubtedly have an effect on sliding at the  $\alpha$ - $\beta$  interface, the contribution of interface morphology to sliding resistance in this case may be minor compared to other factors, which are discussed briefly below.

Plastic deformation will initially occur in the weaker  $\alpha$  phase of both alloys. The strain in the  $\alpha$  and  $\beta$  phases must remain the same to maintain a coherent  $\alpha$ - $\beta$  interface. Initially the  $\beta$  phase will strain elastically in order to maintain coherency at the  $\alpha$ - $\beta$  interface. After substantial deformation in the  $\alpha$  phase, one of two events may happen. The  $\beta$  phase may deform plastically, relieving stress in the  $\alpha$  phase and maintaining coherency at the interface. If the  $\beta$  phase does not deform plastically, coherency at the  $\alpha$ - $\beta$  interface must be lost and interphase interface sliding occurs. Whichever of these deformation mechanisms requires a lower energy will occur. Factors that make plastic deformation of the  $\beta$  phase more difficult will make interface sliding relatively easier.

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Ti-6.0Mn has a  $\beta$  to  $\alpha$  strength ratio of ~4 versus a ratio of ~2.65 for Ti-8.1V. In order to deform the  $\beta$  phase of Ti-6.0Mn, a greater stress is required compared to Ti-8.1V. Likewise, the higher  $\beta$  phase stability of Ti-6.0Mn prevents the formation of stress induced martensite in the  $\beta$  phase. Stress induced martensite does form in the  $\beta$  phase of Ti-8.1V owing to its low stability. The relative ease of stress induced martensite formation in Ti-8.1V makes making interphase interface sliding unnecessary. In contrast, the high strength and stability of the  $\beta$  phase of Ti-6.0Mn precludes stress induced martensite formation, therefore interface interface sliding is easier and will occur.



**Figure 5.3.** High resolution TEM micrograph of the  $\alpha$ - $\beta$  interface of Ti-8.1V, viewed from the  $[000\overline{1}]_{\alpha}$  // $[\overline{1}\overline{1}0]_{\beta}$  direction.



**Figure 5.4.** High resolution TEM micrograph of the  $\alpha$ - $\beta$  interface of Ti-6.0Mn, viewed from the  $[000\overline{1}]_{\alpha}$  // $[\overline{1}\overline{1}0]_{\beta}$  direction.



**Figure 5.5.** High resolution TEM micrograph of the  $\alpha$ - $\beta$  interface of Ti-8.1V, viewed from the  $[1\overline{2}10]_{\alpha}//[1\overline{1}1]_{\beta}$  direction.



**Figure 5.6.** High resolution TEM micrograph of the  $\alpha$ - $\beta$  interface of Ti-6.0Mn, viewed from the  $[1\overline{2}10]_{\alpha} //[1\overline{1}1]_{\beta}$  direction.

#### 5.3.2 Influence of α-β interface morphology on stress induced martensite nucleation

It was suggested above that the higher roughness of the  $\alpha$ - $\beta$  interface in Ti-8.1V may make interface sliding more difficult as compared to Ti-6.0Mn. Roughness at the interface of Ti-8.1V may also contribute to the nucleation of stress induced martensite. Stress induced martensite is known nucleate heterogeneously at interfaces, defects, and other high energy sites [79, 80]. Raj and Ashby discuss accommodation of interface sliding by elastic strain for various interface geometries, which are modeled by different waveforms [51]. The stress in the direction normal to a grain boundary, which is modeled as a sine wave with wavelength  $\lambda$  and amplitude h/2, from an applied shear stress parallel to the boundary varies with position along the boundary and is given by [51]:

$$\sigma_n = -\frac{2}{\pi} \frac{\tau_a \lambda}{h} \sin\left(\frac{2\pi}{\lambda}\right) y \tag{5.1}$$

where  $\tau_a$  is the applied shear stress. Internal stresses are generated normal to features in the surface, and the features with highest amplitude will generate the highest internal stresses. An applied tensile stress on Ti-8.1V which resolves into a shear stress acting on the  $\alpha$ - $\beta$  interface plane will generate normal forces at the interface due to elastic strain and interfacial roughness. These stresses, which are greatest at the peaks of interface features, could assist in the nucleation of stress induced martensite at the  $\alpha$ - $\beta$  interface in Ti-8.1V.

#### 5.3.3 A model for anisotropic interface sliding

It has been observed that sliding occurs anisotropically at interfaces on opposite sides of  $\alpha$  grains in two-phase Ti-Mn alloys. Ankem and Margolin first noted this in their work on Ti-Mn alloys with coarse grained Widmanstätten microstructures with varying amounts of Mn [49], including Ti-5.8wt%Mn with 38%  $\alpha$  phase, which is similar to the Ti-6.0Mn alloy which was examined in this study. Anisotropic interface sliding in Ti-6.0Mn is shown in Figure 5.1 and in Ti-3.9Mn in Figure 5.2.

Ankem [49] proposed that the anisotropic sliding was related to twinning of an interface phase which had been reported in contemporary studies. However, it has been shown that this interface phase does not exist in the bulk, and is an artifact of TEM specimen preparation using electrochemical techniques [66, 67].

In order to explain the observed anisotropic interphase interface sliding, a simple mechanical model based on the locking/unlocking of interfaces due to the formation of growth ledges at  $\alpha$ - $\beta$  interfaces is proposed below. Growth ledges are a common feature of Widmanstätten  $\alpha$ - $\beta$  titanium interfaces. In a review of interphase boundary structures formed during diffusional transformations in titanium alloys Furuhara [89] discusses these growth ledges at length, and provides examples in several titanium alloys with Widmanstätten microstructure. Growth ledges form due to the misfit between phases and are a product of diffusional growth at the  $\alpha$ - $\beta$  interface. They can increase the coherency of the  $\alpha$ - $\beta$  interface, similar to misfit dislocations. The terrace planes of the growth ledges are of the type  $\{\overline{1} \ 100\}_{\alpha} //\{\overline{2} \ 11\}_{\beta}$ , and the spacing of ledges is determined by the misfit [89]. Growth ledges are shown along the  $\alpha$ - $\beta$  interface in single colony crystals of Ti-5Al-2.5Sn-0.5Fe which were tested in creep by Suri et. al. [48].

In the proposed model, shear stresses applied to the  $\alpha$  and  $\beta$  phases which cause sliding would cause locking or unlocking of the interface depending on the applied stress direction and the growth ledge orientation. A schematic showing two possible ledge interface structures that would lead to anisotropic  $\alpha$ - $\beta$  interface sliding is presented in Figure 5.7.

In grains where growth ledges are on one side while the other is smooth, as shown in Figure 5.7 A, sliding could occur on the smooth side A2 while the side with growth ledges A1 locks. If shear stress were instead applied in the opposite directions as shown in Figure 5.7 B then sides B1 and B2 would both slide. In this scenario sliding would occur either uniformly on both sides or on only one side. The microstructural

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configuration of Figure 5.7 A and B has been observed in Ti-6.0Mn, and is shown in Figure 5.8.

Anisotropic interface sliding would also occur if both sides of the  $\alpha$  plate have growth ledges that are mirrored to one another, in which case then one side will lock on the application of shear stresses along the interface while the other slides, as shown in Figure 5.7 C. If the directions of the shear stresses are reversed, then the opposite side will now slide as shown in Figure 5.7 D.

In conclusion, mechanical locking due to growth ledges at the  $\alpha$ - $\beta$  interfaces is a viable model for anisotropic interphase interface sliding in Ti-Mn alloys. This model is supported by experimental observations of anisotropic sliding in Ti-6.0Mn (Figure 5.1) and other Ti-Mn alloys (Figure 5.2) and microstructural observations using TEM show growth ledges in a configuration that would lead to anisotropic sliding in Ti-6.0Mn (Figure 5.8).



**Figure 5.7.** Diagram illustrating how  $\alpha$ - $\beta$  interface structure can affect anisotropic sliding by a locking / unlocking mechanism. In the case of (A) and (B) growth ledges are on one broad face of the Widmanstätten  $\alpha$  plate, and the other is smooth. Under the application of shearing stresses at the interface, A1 will lock while A2 will slide. On application of the opposite shear stress both sides will slide. In the case of (C) and (D) both sides of the  $\alpha$  plate have growth ledges that are on both sides and mirrored. In C, the application of shear stress causes C1 to lock while C2 slides. On application of the opposite shear stress in D, C1 will slide and C2 will lock.



**Figure 5.8.** Bright field TEM composite micrographs showing growth ledges on the broad face of the  $\alpha$ - $\beta$  interface on one side of the  $\alpha$  plate, indicated by arrows. The selected area diffraction patterns from the (a)  $\beta$  phase and (b)  $\alpha$  phase show the specimen is aligned along the  $[0001]_{\alpha}$  // $[110]_{\beta}$  zone axis.

## **5.4.** Conclusions

- 1. The higher  $\beta$  to  $\alpha$  strength ratio, higher  $\beta$  phase stability and smoother  $\alpha$ - $\beta$  interface of Ti-6.0Mn are all factors which contribute to interphase interface sliding, while the ease of stress induced martensite formation and rougher  $\alpha$ - $\beta$  interface precludes interphase interface sliding in Ti-8.1V.
- 2. Stresses generated at the rougher  $\alpha$ - $\beta$  interface of Ti-8.1V may contribute to the formation of stress induced martensite.
- A model is proposed for anisotropic interface sliding by mechanical locking. This model is based on growth ledges at the α-β interface, and is supported by experimental evidence.

#### **Chapter 6**

# Tensile Deformation Mechanisms of α-β Ti-8.1V Alloy with an Equiaxed Microstructure

In the model two-phase Ti-6.0Mn and Ti-8.1V alloys with Widmanstätten  $\alpha$  phase microstructure the tensile deformation mechanisms differed from those observed in single-phase  $\alpha$  and  $\beta$  alloys with compositions matching the phases of the  $\alpha$ - $\beta$  alloys. In the present study the tensile deformation mechanisms of  $\alpha$ - $\beta$  Ti-8.1wt%V with an equiaxed  $\alpha$  phase microstructure were examined. The  $\alpha$  phase of this alloy deformed by slip and twinning, and the  $\beta$  phase was deformed by stress induced hexagonal martensite. The deformation mechanisms of the equiaxed and Widmanstätten alloys were similar. A Burgers orientation relationship has been confirmed for the equiaxed microstructure, therefore the model proposed for  $\alpha$ - $\beta$  interactions in Widmanstätten  $\alpha$ - $\beta$  alloys is applicable to the alloy with equiaxed  $\alpha$  microstructure. In contrast to the Widmanstätten alloy, no extended chains of deformation products were observed because the equiaxed  $\alpha$  grains are not oriented similarly to one another.

#### 6.1. Introduction

Previously, it was found that the tensile deformation mechanisms of the  $\alpha$ - $\beta$  alloy Ti-8.1V with a Widmanstätten  $\alpha$  phase microstructure differed from those of the single phase  $\alpha$  and  $\beta$  alloys with equivalent chemistry. The  $\alpha$  grains with a fine microstructure (<10µm) deformed by slip and twinning, whereas deformation in the single phase  $\alpha$  alloy Ti-1.6V was limited to slip in alloys with fine grains (<62um). The  $\beta$  phase deformed by stress induced hexagonal martensite while the single-phase  $\beta$  alloy Ti-14.8V deformed by slip and twinning. These differences have been modeled in terms of interactions between phases due to a Burgers orientation relationship and the  $\beta$  phase stability. In the current study a Ti-8.1V alloy has been prepared with an equiaxed  $\alpha$  phase microstructure and was tested in tension. The microstructure and deformation mechanisms were examined with a combination of optical microscopy, SEM and TEM.

#### **6.2.** Experimental

Ti-8.1wt%V alloy was prepared with an equiaxed microstructure according to the procedure given in Chapter 2.1. This heat treatment resulted in equiaxed  $\alpha$  phase grains within a  $\beta$  matrix. The average equiaxed  $\alpha$  particle size of 11 µm is comparable to the width of  $\alpha$  grains in the Widmanstätten microstructure analyzed previously.

Tensile specimens were prepared by the method described in Chapter 2.2. Goldpalladium fiducial lines with a 50 µm spacing were deposited on the polished gage surface by the procedure described in Chapter 2.3. These lines are useful for measuring local strain and interface sliding, and are particularly useful in finding the same specimen area for microscopy prior to and following testing.

The surface of the tensile specimen was photographed extensively in several areas prior to testing using an Electroscan ESEM at magnifications of 300x, 1000x and 1500x. The specimen was tensile tested in an Instron 8502 floor model servo-hydraulic testing machine. A clip on extensometer with 12.7 mm gauge length was used to measure strain. The specimen was tested at a strain rate of  $3.28 \times 10^{-5}$  / sec to a total strain of 3%. Following testing the specimen surface was once again photographed using ESEM in the

exact same areas and magnifications used previously. This procedure was used to separate any previously existing features from deformation products that arise during tensile testing. Optical micrographs were taken with a Zeiss ICM 405 inverted metallographic light microscope to image the microstructure and observe microstructure and deformation products over a larger area. Only one tensile test was performed due to the scarcity of material and the high cost and difficulty of specimen preparation.

TEM specimens were prepared from the deformed material in the gage length by the procedure given in Chapter 2.7. A JEOL JEM-4000FX operating at 300 kV was used to examine the microstructure and classify the deformation products.

#### 6.3. Results

The  $\alpha$ - $\beta$  Ti-8.1V alloy with equiaxed microstructure had a yield stress of 619 MPa and showed work hardening. The true stress – true strain plot of this tensile test is provided in Figure 6.1. The optical micrograph in Figure 6.2 shows the morphology of the  $\alpha$  phase microstructure. The majority of the  $\alpha$  grains are equiaxed, and others which have a higher aspect ratio are the result of two or more grains growing together during heat treatment. This micrograph was taken after testing, and close inspection shows stress induced plates in the  $\beta$  phase.

A comparison of the undeformed and deformed microstructures is shown in Figures 6.3 and 6.4 at different magnifications in two areas. In Figure 6.4b, coarse deformation features can be seen crossing from the  $\beta$  phase, through two  $\alpha$  grains, and back into the  $\beta$  phase. The number of plates crossed was generally less than the number of grains traversed in the Widmanstätten alloy.

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**Figure 6.1.** True stress – true strain curve for Ti-8.1V with an equiaxed microstructure. Specimen was tensile tested at room temperature at a strain rate of  $3.28 \times 10^{-5}$  to 3% total strain.



**Figure 6.2.** Optical micrograph of Ti-8.1V with equiaxed  $\alpha$  phase microstructure following testing. The  $\alpha$  phase is black in optical micrographs due to the etchants used.



**Figure 6.3.** SEM micrographs of the surface of the tensile specimen (a) prior to and (b) following tensile deformation. The  $\alpha$  phase appears light and  $\beta$  phase dark in SEM micrographs. Stress induced products are visible in the  $\alpha$  and  $\beta$  phases, indicated by arrows.



**Figure 6.4.** SEM micrographs of the surface of the tensile specimen (a) prior to and (b) following tensile deformation. Stress induced products are visible in the  $\alpha$  and  $\beta$  phases.

The coarse deformation features observed with SEM were analyzed using TEM. Analysis showed that the deformation features were twinning or slip in the  $\alpha$  phase and stress induced hexagonal martensite in the  $\beta$  phase. Figure 6.5 below shows two  $\{10\bar{1}2\}$ twins in the  $\alpha$  phase and a stress induced martensite plate in the  $\beta$  phase. Several more stress induced martensite plates are shown in Figures 6.6 and 6.7. It is noteworthy in Figure 6.6 that the orientation of the martensite plate is nearly the same as the adjacent  $\alpha$ phase. Therefore it is expected that the  $\alpha/\alpha'$  interface will be coherent. In Figure 6.8 slip in the  $\alpha$  phase accompanies a stress induced hexagonal martensite plate in the  $\beta$  phase. Slip was identified as 'a' type prism slip. In this specimen tilt, the electron beam is aligned with  $[1\bar{2}10]_{\alpha}$  // $[1\bar{1}1]_{\beta}$  and dislocations are visible but the diffraction pattern from the stress-induced martensite is from an irrational zone axis. By tilting the specimen nearly 60° in Figure 6.9 to the  $[\bar{1}\bar{1}20]_{\alpha}$  // $[001]_{\beta}$  zone axis, the dislocations are now invisible, and the  $[\bar{2}110]_{\alpha'}$  zone axis of the martensite is aligned with the beam. This martensite plate has a  $\{10\bar{1}1\}$  type twin relationship to the  $\alpha$  phase. Figures 6.8 and 6.9 were taken with the maximum tilts in opposite directions, so this large tilt angle was possible. Usually only one  $\langle 11\overline{2}0 \rangle$  type zone axis is viewable for any  $\alpha$  grain.

Martensite plates in this titanium alloy were observed to cross well-aligned  $\beta$  grain boundaries. This is shown in Figure 6.10, where the interface of two  $\beta$  grains with nearly parallel orientations is crossed by two stress induced martensite plates. In this area it appears that the martensite plates are continuous across the grain boundary, rather than nucleating new martensite plates at the boundary.



**Figure 6.5.** Bright field TEM micrograph showing two  $\{10\overline{1}2\}$  type twins in the  $\alpha$  phase and a stress induced hexagonal martensite ( $\alpha'$ ) plate in the  $\beta$  phase of Ti-8.1V with an equiaxed microstructure. Selected area diffraction patterns are from the (a)  $\beta$  phase (b) stress induced martensite plate (c)  $\alpha$  phase (d) Twin<sub>1</sub>/ $\alpha$  interface and (e) Twin<sub>2</sub>/ $\alpha$  interface. Zone axis is  $[1\overline{1}1]_{\beta}$  // $[1\overline{2}10]_{\alpha}$ .



**Figure 6.6.** Bright field TEM micrograph showing a stress induced hexagonal martensite plate in the  $\beta$  phase of Ti-8.1V. Selected area diffraction patterns from the (a)  $\beta$  phase (b)  $\beta$  / martensite interface and (c) martensite. Zone axis is  $[1\overline{1}1]_{\beta}$  // $[1\overline{2}10]_{\alpha'}$ .



**Figure 6.7.** Bright field TEM micrograph showing two stress induced hexagonal martensite plates, with accompanying selected area diffraction patterns from the (a)  $\beta$  phase and (b) martensite. Zone axis is  $[1\overline{1}1]_{\beta} //[1\overline{2}10]_{\alpha'}$ .



**Figure 6.8.** Bright field TEM micrograph of slip in the  $\alpha$  phase accompanying a stress induced martensite plate in the  $\beta$  phase. SADPs are from the (a)  $\alpha$  phase and the (b)  $\beta$  phase. Zone axis is  $[1\overline{2}10]_{\alpha} //[1\overline{1}1]_{\beta}$  The martensite could not be identified at this tilt.







**Figure 6.10.** Bright field TEM micrograph showing two stress induced hexagonal martensite plates going across a  $\beta$  grain boundary, with accompanying selected area diffraction patterns from (a)  $\beta_1$ , (b)  $\beta_2$  and (c) martensite. Zone axis is approximately  $[110]_{\beta_1} //[110]_{\beta_2} //[24\overline{2}3]_{\alpha'}$ .

## 6.4. Discussion

#### 6.4.1 Similarities between the deformation mechanisms of Ti-8.1V with equiaxed

#### and Widmanstätten microstructure

The  $\alpha$  and  $\beta$  phase deformation mechanisms of the alloy with equiaxed microstructure were the same as those observed in Ti-8.1V with a Widmanstätten microstructure. Hexagonal stress induced martensite was observed in the  $\beta$  phase, which is oriented nearly parallel to the  $\alpha$  phase or is related to  $\alpha$  by a  $\{10\overline{1}1\}$  twin relationship.  $\{10\overline{1}2\}\langle\overline{1}011\rangle$  twins were observed in the  $\alpha$  phase, as well as 'a' type prism slip.

It was not known prior to testing whether the deformation of the equiaxed  $\alpha$ - $\beta$  Ti-8.1V alloy would be similar to the single phase alloys or the Widmanstätten  $\alpha$ - $\beta$  alloy. This was because differences in the microstructure and processing history were expected to alter the Burgers orientation relationship between the  $\alpha$  and  $\beta$  phases, which is present in the Widmanstätten microstructure. Both alloys were extensively worked in the  $\alpha+\beta$ field, which randomizes the  $\alpha$  grain orientation. The Widmanstätten alloy was subsequently heat treated in the  $\beta$  phase, where the  $\alpha$  phase can go into solution. Upon cooling below the  $\beta$  transus, the  $\alpha$  phase nucleates and grows from the  $\beta$  phase, resulting in the plates of Widmanstätten  $\alpha$ . In contrast, the alloy with equiaxed microstructure was kept below the  $\beta$  transus, and so the  $\alpha$  phase should remain in the random orientation it was in following rolling. If the Burgers orientation relationship were absent, deformations mechanisms that are due to interactions dependent on a Burgers orientation relationship would not occur in the equiaxed structure.

The Burgers orientation relationship was, however, observed between the  $\alpha$  and  $\beta$  phase in the alloy with equiaxed microstructure. This relationship is illustrated by the selected area diffraction patterns shown in Figure 6.11. This alloy was heat treated high in the  $\alpha+\beta$  field, close (25 K) to the  $\beta$  transus. At this temperature sufficient diffusion may have occurred to realign the  $\alpha$  phase grains with the  $\beta$  phase. The  $\beta$  phase chemistry and hence stability of the alloys with equiaxed and Widmanstätten microstructures are the same, and  $\omega$  phase is present in the  $\beta$  phase of this alloy as evidenced by well defined  $\omega$  reflections in the diffraction pattern from Figure 6.11b. Therefore, the model based on interactions and  $\beta$  phase stability discussed for Widmanstätten alloys in Chapter 3.1, including elastic interaction stresses,  $\alpha$  phase templating, and the alignment of shear systems for deformation products in the  $\alpha$  and  $\beta$  phases, applies to the alloy with

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equiaxed microstructure, which explains the presence of twins in the  $\alpha$  phase and stress induced martensite in the  $\beta$  phase.



**Figure 6.11.** Bright field TEM micrograph and accompanying selected area diffraction patterns from the (a)  $[0001]_{\alpha}$  and (b)  $[110]_{\beta}$  zone axes showing that the Burgers orientation relationship  $(0001)[1\overline{2}10]_{\alpha}$  //(110) $[1\overline{1}1]_{\beta}$  is obeyed between the  $\alpha$  and  $\beta$  phases of Ti-8.1V with an equiaxed microstructure.

# 6.4.2 Differences between the deformation mechanisms of Ti-8.1V with equiaxed and Widmanstätten microstructure

The major difference in deformation behavior between the two microstructures was that the networks of twins or slip in the  $\alpha$  phase and stress induced martensite in the  $\beta$  phase which alternate across many  $\alpha$  and  $\beta$  grains in the Widmanstätten alloy were not observed in the equiaxed alloy. Such pairing was limited to short distances, observed by SEM as shown in Figure 6.4b and in TEM over a single grain boundary, such as the twins and martensite in close proximity in Figure 4.5 and martensite and slip in Figure 4.8. The microstructural difference between the equiaxed and Widmanstätten alloys affects the scale over which deformation mechanisms can propagate.

It was observed that, although the Burgers orientation relationship was obeyed between that  $\alpha$  and  $\beta$  phases, numerous orientations of the  $\alpha$  phase could be present in close proximity, or even touching one another within a single prior  $\beta$  grain. Figure 6.12 is a composite image of an area where several orientations of the  $\alpha$  phase are present in a single prior  $\beta$  grain. The accompanying selected area diffraction patterns were taken from each grain without tilting. In this small area there are four  $\alpha$  grains with 3 different orientations with respect to the  $\beta$  phase.



**Figure 6.12.** Composite bright field micrograph showing four  $\alpha$  grains with three different orientations within the same  $\beta$  grain.

The numerous  $\alpha$  orientations are a result of the thermomechanical processing. During working in the  $\alpha+\beta$  field the orientation of the grains is randomized. Although subsequent heat treatment may have been sufficient to restore the Burgers orientation for each  $\alpha$  grain, the  $\alpha$  grain would reorient to one of six possible orientations within the  $\beta$ phase. This is in contrast to the Widmanstätten alloy, where the  $\alpha$  grains within a colony have the same orientation [2, 72, 90]. The distance that slip can traverse Widmanstätten plates is limited by the colony size [72, 90]. Likewise, in Ti-8.1V with Widmanstätten microstructure combinations of slip or twins in the  $\alpha$  phase and stress induced martensite in  $\beta$  can traverse many similarly oriented grains. In the equiaxed alloy these deformation mechanisms will be discontinuous between grains of unlike orientation. The yield stress of the alloy with equiaxed microstructure is slightly higher than that of the alloy with Widmanstätten microstructure, 619 MPa vs. 597 MPa. The lack of long-range order with respect to  $\alpha$  grain orientations forms barriers to the transmission of slip, twins and martensite across many grains, which may contribute to the a higher yield stress of the equiaxed microstructure. These barriers to the transmission of deformation mechanisms should also result in a higher creep resistance in alloys with an equiaxed microstructure.

In addition, the prior  $\beta$  grains appeared much smaller in the equiaxed alloy, with frequent  $\beta$  grain boundaries and changes in  $\beta$  phase orientation. This grain size was not measured, but prior  $\beta$  grain boundaries were observed much more frequently during TEM observation than they were for the Widmanstätten alloy. This is due to the lower heat treatment temperature, which stayed below the  $\beta$  transus temperature which did not allow for as much  $\beta$  grain growth. So in addition to the multiple orientations of  $\alpha$  within each  $\beta$ grain, the  $\beta$  orientation may change more frequently, although adjacent  $\beta$  grains can be oriented similarly as shown in Figure 4.10.

#### 6.5. Conclusions

- 1. Ti-8.1V with an equiaxed microstructure has a yield strength of 619 MPa, and deformed by slip and twinning in the  $\alpha$  phase and by stress induced hexagonal martensite in the  $\beta$  phase. These deformation mechanisms are similar to those observed in Ti-8.1V with a Widmanstätten microstructure.
- 2. A Burgers orientation relationship exists between the  $\alpha$  and  $\beta$  phases of Ti-8.1V with an equiaxed microstructure. Therefore, the interactions between phases discussed for the Widmanstätten alloys in Chapter 3 can also apply to this microstructure, and explain the presence of twinning and stress induced martensite.
- 3. There were no extended chains of twins or slip in  $\alpha$  and martensite in  $\beta$ , although these could be in combination locally. This is due to frequent changes in  $\alpha$  phase orientation within the  $\beta$  grains in close proximity to one another, which act as a barrier for the transmission of stress.

#### Chapter 7

# Influence of the ω Phase on the Tensile and Creep Deformation Mechanisms of α-β Titanium Alloys

As discussed in Chapters 3, 4, and 6, it was observed that stress induced hexagonal martensite ( $\alpha'$ ) forms in the  $\beta$  phase of the  $\alpha$ - $\beta$  alloy Ti-8.1V with Widmanstätten and equiaxed microstructures. This has been attributed to the stability of the  $\beta$  phase and interactions between the  $\alpha$  and  $\beta$  phases. In this investigation the role of the nanocrystalline  $\omega$  phase on the formation of  $\alpha'$  in two-phase titanium alloys is examined. It was determined that the  $\alpha$ - $\omega$  interactions play a role in the nucleation of  $\alpha'$ . The  $\alpha$ - $\omega$  misfit strain places stress on  $\omega$  planes that assist the  $\omega \rightarrow \alpha'$  transformation. The misfit strain is reduced by transformation of  $\omega$  to  $\alpha'$ , which has a  $\{10\overline{1}1\}$ twin relationship to the adjacent  $\alpha$  phase. Shear stresses from slip and twinning in the  $\alpha$  phase act on the  $\omega \rightarrow \alpha$  transformation shear systems. TEM and HREM were used to measure the  $\alpha$  and  $\omega$ lattice parameters, determine the orientation relationships of  $\alpha$ ,  $\beta$ ,  $\omega$ , and  $\alpha'$  and observe the  $\alpha$ - $\omega$  and  $\alpha$ - $\alpha'$  interfaces.

#### 7.1. Introduction

It was observed previously that the  $\beta$  phase of the two-phase alloy Ti-8.1V deforms in tensile and creep tests by stress induced hexagonal martensite ( $\alpha'$ ), while the single phase  $\beta$  alloy with the same composition, Ti-14.8V, deforms by slip and  $\{332\}\langle113\rangle$  twinning. The difference in deformation mechanisms has been attributed to

the presence of the  $\alpha$  phase. Influences of the  $\alpha$  phase include elasto-plastic interactions, the  $\alpha$  phase acting as a template for hexagonal martensite, and resolved shear stresses from  $\alpha$  phase slip and twinning systems acting on shear systems for the  $\beta \rightarrow \alpha'$ transformation. In this investigation, consideration is given to the effect of the interactions between the  $\alpha$  phase and the nanocrystalline  $\omega$  phase on the  $\beta$  phase deformation mechanisms.

In  $\beta$  titanium alloys the formation of the  $\omega$  phase is dependent upon the alloy stability. In Ti-V alloys, athermal  $\omega$  phase will form in alloys with V contents up to about 20wt%V upon quenching [6,32]. The vanadium content of the  $\beta$  phase of Ti-8.1V is 14.8wt%V. Therefore significant  $\omega$  phase is expected, and is observed in selected area diffraction patterns and dark field images of the  $\beta$  phase of Ti-8.1V.

The  $\alpha$  phase has a hexagonal closed packed (HCP) structure, and the  $\beta$  phase has a body centered cubic (BCC) structure. The  $\omega$  phase, which exists as a nanostructured phase within the beta phase of Ti-8.1V, has a P6/mmm hexagonal structure (Strukturbericht C32 designation) with alternating layers of hexagonal sheets [7,11,12]. Although the  $\alpha$  and  $\omega$  phases are both hexagonal, their crystal structures are quite different,  $\alpha$  having a c/a ratio of 1.60 and  $\omega$  having a c/a ratio of 0.61.

There is a well defined orientation relationship between the  $\alpha$  and  $\beta$  phase in titanium alloys with a Burgers orientation relationship. When the  $\omega$  phase is present, there is an orientation relationship between four different orientations of the  $\omega$  phase and the  $\beta$  phase [17]. The orientation relationship between the  $\alpha$  and  $\beta$  phases is:

$$\alpha$$
:  $(0001) [1\overline{2}10]_{\alpha} //(110) [1\overline{1}1]_{\beta}$ 

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Four variants of the  $\omega$  phase have orientation relationships with the  $\beta$  phase:

$$\omega_{1}: (\overline{1} 010)[0001]_{\omega_{1}} //(\overline{1} 12)[1 \overline{1} 1]_{\beta}$$
  

$$\omega_{2}: (\overline{1} 010)[0001]_{\omega_{2}} //(\overline{1} \overline{1} 2)[\overline{1} 11]_{\beta}$$
  

$$\omega_{3}: (\overline{1} 010)[0001]_{\omega_{3}} //(\overline{1} \overline{1} 2)[111]_{\beta}$$
  

$$\omega_{4}: (\overline{1} 010)[0001]_{\omega_{4}} //(112)[\overline{1} \overline{1} 1]_{\beta}$$

These relationships have been confirmed by selected area diffraction from the  $[0001]_{\alpha}//[110]_{\beta}$  zone axes of Ti-8.1V with Widmanstätten and equiaxed microstructures. The diffraction patterns for two orientations of the  $\omega$  phase are visible in any given diffraction pattern from the <110>  $\beta$  zone axis. The  $[110]_{\beta}$  pattern contains patterns from the  $[\overline{1}2\overline{1}0]_{\omega_1}$  and  $[\overline{12}10]_{\omega_2}$  zone axes. These diffraction patterns are presented below in Figure 7.1. The orientation relationships of  $\alpha$ ,  $\beta$ , and  $\omega_1$  and  $\omega_2$  are shown schematically in Figure 7.2.



**Figure 7.1.** Selected area diffraction patterns of the (a)  $\alpha$  phase oriented along  $[0001]_{\alpha}$  and the (b)  $\beta$  phase oriented along the  $[110]_{\beta}$  zone axis. The diffraction patterns of  $\omega_1$  and  $\omega_2$  oriented along  $[\overline{1}2\overline{1}0]_{\omega_1}$  and  $[1\overline{2}10]_{\omega_2}$  are contained within the  $\beta$  phase pattern. The above diffraction patterns were taken from adjacent grains without tilting the specimen.



**Figure 7.2.** Projections of the  $(0001)_{\alpha}$ ,  $(0001)_{\beta}$ , and  $(\overline{1} 2 \overline{1} 0)_{\omega 1}$  and  $(1\overline{2} 10)_{\omega 2}$  planes showing the orientation relationship of the  $\alpha$ ,  $\beta$ , and  $\omega$  phases. Plane traces are perpendicular to the projection plane, and are drawn as lines, with line intersections indicating atomic positions. The blue lines are traces of the  $\alpha/\beta$  interface planes. Illustration is to scale.

Due to these well defined orientation relationships, interactions can occur between the  $\alpha$ ,  $\beta$  and  $\omega$  phases. The present work focuses on how  $\alpha/\omega$  and  $\beta/\omega$ interactions affect the  $\beta$  phase deformation mechanisms in  $\alpha$ - $\beta$  titanium alloys. Although Ti-8.1V is used as the model system in this work, these results are applicable to any twophase titanium alloy with a Burgers orientation relationship between the  $\alpha$  and  $\beta$  phases when  $\omega$  phase is present within the  $\beta$  phase.

#### 7.2. Experimental

#### 7.2.1 TEM and HREM

Specimens were prepared from Ti-8.1V with a Widmanstätten microstructure by the procedure described in Chapter 2.7. A JEOL JEM-4000FX operating at 300KeV was used to obtain the images and diffraction patterns for this study. In order to view the  $\omega$ phase and ensure that the HREM images were taken without any overlap of the  $\alpha$  and  $\beta$ phases the sample was tilted along the (0001)<sub> $\alpha$ </sub> and (110)<sub> $\beta$ </sub> zone axes. As shown in Figures 7.1 and 7.2, the  $\alpha$ - $\beta$  interface plane is parallel to the beam direction in this specimen orientation, and two orientations of the  $\omega$  phase are visible. In order to reveal the  $\alpha/\beta/\alpha'$ orientation relationship, the specimen was positioned with the beam parallel to  $[1 \overline{1} 1]_{\beta} //[1 \overline{2} 10]_{\alpha}$ . In this orientation the beam is parallel to the  $\alpha/\alpha'$  interface plane, and the diffraction patterns of both the  $\alpha$  phase and  $\alpha'$  are clearly visible. High resolution microscopy of the  $\alpha/\alpha'$  twinned interface can only be performed in areas where the interface lies in the correct orientation very close to the foil edge, and in the correct orientation, which is extremely rare and difficult to locate.

#### 7.2.2 Calculation of resolved shear stresses from α phase deformation products

The  $\alpha$  and  $\omega$  phase coordinate systems were converted to the  $\beta$  phase coordinate system using the orientation relationships above and using the procedure described in Chapter 2.10. The conversion matrices are available in Appendix H. The directions and planes important for slip and twinning in  $\alpha$  and for the martensitic transformation in  $\omega$ were then converted to equivalent vectors in the  $\beta$  phase coordinate system, which are available in Appendix I. Calculation of the resolved shear stresses from slip and twinning in the  $\alpha$  phase on shear systems in the  $\omega$  phase was then possible using this common coordinate system. The  $\beta$  phase coordinate system is convenient not only because of the orientation relationships with the  $\alpha$  and  $\omega$  phases, but also because calculations are easily performed in the cubic system. Calculation of the resolved shear stresses from 'a' type  $(\vec{b} = 1/3\langle 11\overline{2}0 \rangle)$  prism and basal slip and  $\{10\overline{1}1\}$  and  $\{10\overline{1}2\}$  type twins in the  $\alpha$  phase on the  $\omega$  to  $\alpha'$  shear systems was performed by tensor transformation as described in Chapter 2.10.

#### 7.3. Results and Discussion

#### 7.3.1 α-ω interface

The  $\omega$  phase is present in high concentrations in the  $\beta$  phase of Ti-8.1V. The exact concentration is difficult to determine due to the multiple orientations and the small particle size in relation to the sample thickness. A dark field image of nanocrystalline athermal  $\omega$  phase within the  $\beta$  of Ti-8.1V using a single  $\omega$  reflection is shown in Figure 7.3. Only one of four orientations of the  $\omega$  phase is shown when imaged in this manner. The bright areas are  $\omega$  phase.

Due to the high concentration of  $\omega$  phase, it seems reasonable that along the  $\alpha$ - $\beta$  interface there will be a great number of  $\alpha$ - $\omega$  interfaces. This was confirmed with HREM images of the interface. Orientations 1 and 2 of the  $\omega$  phase were observed in proximity to the  $\alpha$  phase. Figure 7.4 shows the interface of the  $\alpha$  phase with a region of  $\omega_1$ , and Figure 7.5 shows the  $\alpha$  phase nearly in contact with  $\omega_2$ .


**Figure 7.3.** Dark field TEM micrograph of Ti-8.1wt%V alloy. Bright spots are nanocrystalline  $\omega$  phase particles within the  $\beta$  phase.



**Figure 7.4.** HREM image of the  $\alpha$ - $\omega$ - $\beta$  interface of Ti-8.1V with accompanying selected area diffraction patterns. The  $\omega$  phase shown is orientation 1. Notice the lattice distortion of the  $\alpha$  and  $\omega$  phases at the interface. Inset selected area diffraction patterns show the orientation of the specimen to the electron beam of  $[0001]_{\alpha} //[110]_{\beta} //[\overline{1}2\overline{1}0]_{\omega_1}$ .



**Figure 7.5.** – HREM of the  $\alpha$ - $\omega$ - $\beta$  interface of Ti-8.1V with accompanying selected area diffraction patterns. The  $\alpha$  phase is nearly in contact with a region of  $\omega_2$ . Inset selected area diffraction patterns show the orientation of the specimen to the electron beam of  $[0001]_{\alpha} //[110]_{\beta} //[1\overline{2}10]_{\omega_2}$ .

#### 7.3.2 $\omega$ $\rightarrow$ hexagonal martensite ( $\alpha$ ') transformation

The  $\alpha$  to  $\omega$  phase transformation is a reversible martensitic transformation, where  $\alpha$  is transformed to  $\omega$  under hydrostatic stress [7-10]. Therefore, the  $\alpha \rightarrow \omega$  and  $\omega \rightarrow \alpha$  transformation pathways are equivalent. According to the work of Silcock [11], the  $\alpha$  phase is transformed to the  $\omega$  phase by a series of shears of  $(1 \overline{1} 00)_{\alpha}$  planes in the

 $[11\overline{2}0]_{\alpha}$  direction. This results in a transformation where the previous  $(0001)_{\alpha}$  plane is now a  $(11\overline{2}0)_{\omega}$  plane. The reverse transformation takes place by shear on the  $(1\overline{1}00)_{\omega}$ planes in the  $[11\overline{2}0]_{\omega}$  direction, resulting in  $(0001)_{\alpha}$  planes parallel to the previous  $(11\overline{2}0)_{\omega}$  planes. In this way each  $\omega$  crystal can transform into three orientations of  $\alpha$  with the c axis oriented 60° from one another.

There are other transformation pathways presented for the  $\alpha \leftrightarrow \omega$  transformation [7, 9, 10], but these are not relevant for  $\omega$  phase that is contained within the  $\beta$  phase. Instead, these studies are concerned with  $\omega$  phase that is transformed from in HCP metals (Ti and Zr) under high hydrostatic pressures at elevated temperatures, resulting in bulk  $\omega$  or  $\omega$  within  $\alpha$ . In such a case the transformations would not maintain a Burgers orientation relationship between the transformed  $\alpha'$  and the surrounding  $\beta$  phase, which has been observed in Ti-8.1V and is discussed below. Therefore the shear transformation proposed by Silcock is used for this analysis.

#### 7.3.3 Resulting $\alpha/\alpha'$ orientation

The four orientations of  $\omega$  phase are each aligned such that their basal ( $\{0001\}_{\omega}$ ) planes are parallel to the  $\{111\}_{\beta}$  type planes in the  $\beta$  phase, and  $\{11\overline{2}0\}_{\omega}$  planes are parallel to  $\{110\}_{\beta}$  planes. During the transformation from  $\omega$  to  $\alpha'$  the  $\{11\overline{2}0\}_{\omega}$  planes transform to  $\{0001\}_{\alpha'}[8, 11]$ . This means that any one of the four orientations of  $\omega$  can transform into three independent orientations of  $\alpha'$ . The resulting orientations of this transformation are illustrated schematically in Figure 7.6. Due to the  $\{11\overline{2}0\}_{\omega}//\{110\}_{\beta}$  orientation of  $\omega$  to the  $\beta$  phase, all of these  $\alpha'$  plates obey the Burgers orientation relationship with the  $\beta$ 

phase. Martensite that results from the  $\omega_1$  and  $\omega_2$  orientations of  $\omega$  will either have the same orientation as the  $\alpha$  phase,  $(0001)_{\alpha'}$  // $(0001)_{\alpha}$  or  $(0001)_{\alpha'}$  will be rotated 60° with respect to  $(0001)_{\alpha}$ . In martensite which results from the  $\omega_3$  and  $\omega_4$  orientations,  $(0001)_{\alpha'}$  will be rotated 90° or 60° with respect to  $(0001)_{\alpha}$ . The 60° rotation is nearly equivalent to a  $\{10\overline{1}1\}$  twin relationship with  $\alpha$ , which has a rotation between basal planes calculated as 58° in Ti-8.1V. This angle may change slightly in other alloys due to differences in the c/a ratio. The 90° rotation would be close to the 94° rotation of a  $\{10\overline{1}2\}$  twin. The  $\{10\overline{1}1\}$  near-twin relationship has been observed between stress induced martensite and the  $\alpha$  phase of Ti-8.1V, as discussed in Chapters 3, 4 and 6, and is shown by the selected area diffraction patterns in Figure 7.7. In cases where martensite plates form from the omega phase in different orientations within a single  $\beta$  grain, each plate will have a twinned relationship to the  $\alpha$  phase, and both martensite plates will obey the Burgers orientation relationship with the  $\beta$  phase, as shown in Figure 7.8. Therefore, the interface created between the  $\alpha$  phase and a stress induced hexagonal martensite plate should be coherent, with either a matching or twin-like interface. The coherency of the twin-like interface was confirmed using HREM, and is shown in Figure 7.9. This is in contrast to the  $\alpha$ - $\omega$  interface that exists prior to the  $\omega \rightarrow \alpha'$  transformation, which is discussed below.



**Figure 7.6.** Schematic of the  $\omega$  to hexagonal martensite transformation, which can result in three different orientations of martensite for each orientation of  $\omega$ . Because the  $\{0001\}_{\alpha'}$  planes of martensite form parallel to  $\{\overline{12}10\}_{\omega}$  planes [11], one orientation of martensite will have the same orientation as the  $\alpha$  phase, and the other two will be rotated 60° with respect to the  $\alpha$  phase.



**Figure 7.7.** Selected area diffraction pattern taken from (a) a stress induced martensite plate in the  $\beta$  phase and (b) the adjacent  $\alpha$  phase. The composite pattern is shown in (c), showing a near  $\{10\overline{1}1\}$  twin relationship and mirror symmetry over the  $(10\overline{1}1)_{\alpha}$  plane.



**Figure 7.8.** Bright field TEM micrograph showing two stress-induced hexagonal martensite plates ( $\alpha'$ ) within the  $\beta$  phase of Ti-8.1V tested in tension. Accompanying diffraction patterns show that both martensite planes have a  $\{10\overline{1}1\}$  near twin relationship to the  $\alpha$  phase, and that the martensite forms with  $\{0001\}_{\alpha'}$  planes parallel to  $\{110\}_{\beta} //\{11\overline{2}0\}_{\omega}$  planes.



**Figure 7.9.** High resolution TEM micrograph showing the interface of stress induced martensite,  $\alpha'$ , and the  $\alpha$  phase. Notice the  $\{10\overline{1}1\}$  near twin relationship between the phases. Selected area diffraction patterns from each phase and the interface taken parallel to the  $(1\overline{2}10)$  zone axis are inset.

#### 7.3.4 α-ω misfit strain

As discussed above, the  $\alpha'/\alpha$  interface is coherent. This is in contrast to the observed misfit at the  $\alpha/\omega$  interface. Both the  $\alpha$  and  $\omega$  phases at the interface appear distorted due to misfit strain.

The misfit between the  $\alpha$  and  $\omega_1$  and  $\omega_2$  phases has been calculated for orientations 1 and 2 of  $\omega$  along the  $\alpha/\omega$  interface parallel to the prior  $\alpha$ - $\beta$  interface. This interface is important because of the nature of  $\alpha$  and  $\omega$  formation in the Widmanstätten alloys. When the alloy is cooled below the  $\beta$  transus temperature, the  $\alpha$  phase grows from the  $\beta$  phase, resulting in a fixed  $\alpha/\beta$  interface plane of  $(\overline{5} 140)_{\alpha}//(\overline{3} 34)_{\beta}$  given by the Burgers orientation relationship described above and illustrated in Figure 7.2. The Burgers orientation relationship was also observed in Ti-8.1V with an equiaxed microstructure, so this analysis applies to both microstructures. When the alloys are quenched from 963 K, athermal  $\omega$  phase nucleates throughout the  $\beta$  phase. When the  $\omega$ phase forms at the  $\alpha/\beta$  interface the interface plane remains unchanged, resulting in an  $\alpha/\omega$  interface plane that is parallel to the prior  $\alpha/\beta$  interface. These planes have been calculated as  $(20\overline{2}7)_{\omega_1}$  and  $(\overline{1} 01 12)_{\omega_2}$  for  $\omega_1$  and  $\omega_2$  respectively.

Strain has been calculated along the  $[0001]_{\alpha}$  and  $[1\overline{3}20]_{\alpha}$  directions, which are perpendicular directions in the  $\alpha/\omega$  interface plane. Strain in the direction parallel to  $[0001]_{\alpha}$  in  $\omega_1$  or  $\omega_2$  is given by Equation 7.1:

$$\varepsilon_{\omega 1,\omega 2} = \frac{c_{\alpha} - a_{\omega}}{a_{\omega}} \tag{7.1}$$

This strain is minimal for both  $\omega_1$  and  $\omega_2$  due to the similarly of the  $\{110\}_{\beta}$  and  $\{11\overline{2}0\}_{\omega}$  planes. The misfit strain between near-parallel  $\alpha$  and  $\omega$  planes in a direction parallel to the  $[1\overline{3}20]_{\alpha}$  direction are greater than those in the  $[0001]_{\alpha}$  direction. The best matching planes are  $(0\overline{1}10)_{\alpha} / (0001)_{\omega_1}$  and  $(1\overline{2}10)_{\alpha} / (\overline{1}010)_{\omega_2}$ . The strains in the  $[1\overline{3}20]_{\alpha}$  direction are given by Equations 7.2 and 7.3:

$$\varepsilon_{\omega 1} = \frac{\frac{2\sqrt{21}}{9}a_{\alpha} - \frac{\sqrt{(7c_{\pi})^{2} + \left(\frac{\sqrt{3}}{2}a_{\omega}\right)^{2}}}{7}}{\frac{\sqrt{(7c_{\pi})^{2} + \left(\frac{\sqrt{3}}{2}a_{\omega}\right)^{2}}}{7}}$$
(7.2)
$$\varepsilon_{\omega 2} = \frac{\frac{2\sqrt{21}}{9}a_{\alpha} - \frac{\sqrt{c_{\pi}^{2} + \left(\frac{6\sqrt{3}}{2}a_{\omega}\right)^{2}}}{6}}{\frac{\sqrt{c_{\pi}^{2} + \left(\frac{6\sqrt{3}}{2}a_{\omega}\right)^{2}}}{6}}$$
(7.3)

The measured lattice parameters for  $\alpha$  and  $\omega$  in Ti-8.1V using selected area diffraction patterns are presented in Table 7.1, and the calculated misfit strains in both directions using the above equations are presented in Table 7.2.

Phase	a (nm)	c (nm)
α	0.2937	0.4699
ω	0.4688	0.2858

**Table 7.1.** Lattice Parameters for the  $\alpha$  and  $\omega$  phases

	% Strain in $\omega$ - Direction	
Interface	$[0001]_{\alpha}$	$\left[1\overline{3}20\right]_{\alpha}$
$\alpha/\omega_1$	0.245	2.584
$\alpha/\omega_2$	0.245	-26.8190

**Table 7.2.** Misfit strain between the  $\alpha/\omega_1$  and  $\alpha/\omega_2$  interfaces.

The interface strain acts to expand both  $\omega_1$  and  $\omega_2$  along their [0001] direction, by placing a tensile stress on the  $(\bar{1}010)$  plane of  $\omega_1$  in the [0001] direction and a compressive stress on the (0001) plane in the  $[\bar{1}010]$  direction in  $\omega_2$ . Silcock [11] states that an expansion of ~4% in the [0001]<sub> $\omega$ </sub> direction is required to give coincidence between  $\alpha$  and  $\omega$  phases in the transformation. Silcock also states that a ~2% expansion is required in the  $[1\bar{2}10]_{\omega}$  direction. The strain from the  $\alpha$  phase in the  $\langle 1\bar{2}10 \rangle_{\omega}$  directions of  $\omega_1$  and  $\omega_2$  parallel to  $[0001]_{\alpha}$  is ~0.25%, and places a tensile stress on the  $\omega$  phases for this required expansion. Therefore, in addition to being a high energy interface, the interfacial misfit places stresses on the  $\omega$  phase.

The interface between  $\alpha$  and  $\omega$  has a high interfacial free energy due to the high misfit strain. The free energy is lowered by the formation of stress induced hexagonal martensite, as the new interface will be a coherent twin-like interface. The strain energy reduction at the  $\alpha/\omega$  interface is no doubt a driving force in the  $\omega$  to  $\alpha'$  transformation.

The application of misfit stresses provides a driving force in the  $\omega$  to  $\alpha$  transformation, and forming a coherent twin-like interface between  $\alpha$  and  $\alpha'$  reduces the

interfacial free energy of the strained  $\alpha/\omega$  interface. In these ways the  $\alpha$  phase acts as a template for the formation of hexagonal martensite, which was proposed in Chapter 3.

# 7.3.5 Calculations of resolved shear stress from slip, twinning in $\alpha$ on $\omega \rightarrow \alpha'$ shear systems

It is known that there is an alignment of slip systems in the  $\alpha$  and  $\beta$  phases due to the Burgers orientation relationship [1, 36, 41, 72-74]. In Chapter 3 it was shown that the magnitude of resolved shear stress from  $\alpha$  phase deformation mechanisms, slip and twinning, was significant on shear systems for the  $\beta \rightarrow \alpha'$  transformation. In this chapter the magnitudes of resolved shear stress have been calculated for  $\alpha$  phase deformation mechanisms on the  $\omega \rightarrow \alpha$  transformation shear systems discussed in Section 2 above.

Stress induced martensite plates in the  $\beta$  phase of Ti-8.1V were accompanied by either slip or twinning in the adjacent  $\alpha$  phase. Previously, calculations have been performed to determine the resolved shear stresses from  $\alpha$  phase slip or twinning on the shear systems for twinning or stress induced martensite formation in  $\beta$ . The  $\omega$  phase is an intermediate phase in the  $\beta$  to  $\alpha$  phase transformation [17]. It therefore stands to reason that shear stresses acting on the  $\omega$  phase at the  $\alpha$ - $\omega$  interface would nucleate martensite even more readily than the same shear stresses acting on the  $\beta$  phase. Contact of  $\omega$  with the  $\alpha$  phase transmits this stress directly to the  $\omega \rightarrow \alpha'$  shear systems. In this investigation the resolved shear stress from prism slip, basal slip, and  $\{10\overline{1}1\}$  and  $\{10\overline{1}2\}$  twins in the  $\alpha$ phase on the  $\omega \rightarrow \alpha'$  shear transformation systems have been calculated.

Three independent prism slip systems and six independent twinning systems for both  $\{10\overline{1}1\}$  and  $\{10\overline{1}2\}$  type twins were considered for deformation products observed in the  $\alpha$  phase. Slip was observed in Ti-8.1V with 'a' type screw dislocations, therefore Burgers vectors of  $1/3\langle 11\overline{2}0\rangle$  were used in the calculations. Because 'a' type screw dislocations can also slip on the basal plane in titanium, the three independent basal slip systems were also included in these calculations. The shear stress from these  $\alpha$  phase deformation mechanisms act in varying degrees on the three independent  $\omega \rightarrow \alpha'$ transformation shear systems in each of the four  $\omega$  phase orientations. All possible combinations of resolved shear stress have been considered. The maximum resolved shear stresses on the  $\omega$  phase transformation systems are presented below in Tables 7.3, 7.4, 7.5 and 7.6. Any negative values of resolved shear stress are a result of only independent shears being used in the calculations. The absolute values should be considered, as negative values can be made positive by simply considering shear in the opposite direction along the same shear plane.

		P	rism Slip Systen	n
$\omega$ orientation	$\omega$ shear system	$(1\overline{1}00)[11\overline{2}0]$	$(\overline{1}010)[1\overline{2}10]$	$(0\overline{1}10)[\overline{2}110]$
1	(1010)[0001]	-0.51	1	0.49
2	(1010)[0001]	0.15	0.78	0.93
3	$(0\overline{1}10)[0001]$	-0.53	0.38	-0.14
4	(1 1 00)[0001]	0.53	-0.38	0.14

**Table 7.3.** Maximum resolved shear stress from prism slip onto  $\omega \rightarrow \alpha$  shear systems

		E	Basal Slip Syster	n
$\omega$ orientation	$\omega$ shear system	$(0001)[11\overline{2}0]$	$(0001)[1\overline{2}10]$	$(0001)[\overline{2}110]$
1	$(0\overline{1}10)[0001]$	0.41	-0.87	0.42
2	$(0\overline{1}10)[0001]$	-0.58	-0.29	0.85
3	$(0\overline{1}10)[0001]$	-0.08	0.67	-0.57
4	$(0\overline{1}10)[0001]$	0.25	0.48	-0.71

**Table 7.4.** Maximum resolved shear stress from basal slip onto  $\omega \rightarrow \alpha$  shear systems

**Table 7.5.** Maximum resolved shear stress from  $\{10\overline{1}1\}$  twins onto  $\omega \rightarrow \alpha$  shear systems

		Twin Shear System		
$\omega$ orientation	$\omega$ shear system	$(\overline{1}011)[10\overline{1}2]$	$(10\overline{1}1)\overline{1}012$	$(0\overline{1}11)[01\overline{1}2]$
1	(1 1 00)[0001]	0.00	0.01	-0.22
2	(1 1 00)[0001]	0.58	-0.31	0.18
3	(1010)[0001]	-0.50	-0.80	-0.56
4	(1010)[0001]	-0.8	-0.5	-0.89
		Twin S	hear System (con	ntinued)
$\omega$ orientation	$\omega$ shear system	$(01\overline{1}1)[0\overline{1}12]$	$(\overline{1}101)[1\overline{1}02]$	$(1\overline{1}01)[\overline{1}102]$
1	$(1\overline{1}00)[0001]$	0.59	0.22	-0.59
2	$(1\overline{1}00)[0001]$	-0.01	0.16	-0.57
3	$(\overline{1}010)[0001]$	-0.89	-0.40	-0.38
	\ / <b>L</b> _			

		Twin Shear System		
$\omega$ orientation	$\omega$ shear system	$(\overline{1}012)[10\overline{1}1]$	$(10\overline{1}2)\overline{1}011$	$(0\overline{1}12)[01\overline{1}1]$
1	(1010)[0001]	0.01	0.01	-0.45
2	(1010)[0001]	-0.32	-0.32	-0.20
3	(1010)[0001]	-0.79	-0.75	-0.89
4	(1010)[0001]	-0.75	-0.79	-0.84
		Twin S	hear System (coi	ntinued)
$\omega$ orientation	$\omega$ shear system	$(01\overline{1}2)[0\overline{1}11]$	$(\overline{1}\ 102)[1\ \overline{1}\ 01]$	$(1\overline{1}02)[\overline{1}101]$
1	(1010)[0001]	-0.45	0.45	0.45
2	(1010)[0001]	-0.20	0.50	0.50
3	(1010)[0001]	-0.84	-0.46	-0.47
4	(1010)[0001]	-0.89	-0.46	-0.46

**Table 7.6.** Maximum resolved shear stress from  $\{10\overline{1}2\}$  twins onto  $\omega \rightarrow \alpha$  shear systems

The results of the above calculations have several consequences. First, it should be noted that the resolved shear stress from  $\alpha$  phase slip and twins acts more strongly on the  $\omega$  phase shear systems than  $\beta$  phase twinning systems (maximum values presented in Chapter 3, full calculations in Appendix J). In the few instances where the resolved shear stress is greater on a twinning shear system, the magnitude of resolved stress on the  $\beta$ twinning system is low, and therefore the twinning will not occur. Therefore, stress induced martensite is likely to form due to the combination of applied stress and the resolved shear stress of slip and twinning on the  $\omega$  phase.

Second, the  $\omega$  orientations that will transform are different depending on whether slip or twinning is the primary deformation mechanism in the adjacent  $\alpha$  phase. In the case of 'a' type prism or basal slip in  $\alpha$ ,  $\omega$  orientations 1 or 2 are more likely to transform. For  $\{10\overline{1}1\}$  or  $\{10\overline{1}2\}$  twinning in  $\alpha$ ,  $\omega$  orientations 3 or 4 will transform. The values of highest resolved shear stress for each  $\alpha$  slip or twinning system are in bold to highlight this point.

Third, within each orientation there is a preferred transformation shear system. Although three possible martensite orientations are possible, it is most likely that one of these will form preferentially within any  $\beta$  grain. This means that multiple martensite plates within a  $\beta$  grain will have the same orientation relationships to one another, as well as the  $\beta$  and  $\alpha$  phases, which has been observed experimentally. There are exceptions in cases where, for a given slip or twinning system there are more than one  $\omega \rightarrow \alpha'$  transformation system with equivalent resolved shear stress. This is the case for the  $(\overline{1}011)[10\overline{1}2]$  twin system, which acts on  $(\overline{1}010)[0001]$  and  $(0\overline{1}10)[0001]$  of  $\omega_4$  approximately equally and for the  $(0001)[1\overline{2}10]$  basal slip system acting on  $(0\overline{1}10)[0001]$  and  $(1\overline{1}00)[0001]$  of  $\omega_1$ . Such an occurrence would explain martensite plates forming in two directions in a single  $\beta$  grain as shown in Figure 7.7.

#### 7.3.6 Martensite growth through ω-β interactions

It was shown above how  $\alpha/\omega$  phase interactions can aid in the nucleation of stress induced hexagonal martensite from the  $\omega$  phase within the  $\beta$  phase of  $\alpha$ - $\beta$  titanium alloys with a Widmanstätten microstructure. The question as to how the martensite continues to propagate through the  $\beta$  phase must be considered. The answer lies in the orientation relationship of the  $\omega$  and  $\beta$  phases. The orientation relationship

 $\{\overline{1}\ 010\}(0001)_{\omega}$  // $\{\overline{1}\ 12\}(1\ \overline{1}\ 1)_{\beta}$  means that each shear system for a  $\omega \rightarrow \alpha'$  transformation is exactly parallel to a  $\beta \rightarrow \alpha'$  shear system as given by Otte [74]. The transformed  $\alpha'$  from the  $\beta$  phase will have the same orientation as  $\alpha'$  from the  $\omega$  phase. Therefore, once the

transformation is initiated in the  $\omega$  phase, it can continue through the  $\beta$  phase along parallel shear systems.

#### 7.3.7 $\omega$ phase in the single-phase $\beta$ alloy Ti-14.8V

The role of the  $\omega$  phase in the formation of stress induced hexagonal martensite in two phase titanium alloys, particularly Ti-8.1V, has been discussed at length above. During this transformation the  $\omega$  and  $\beta$  phases are consumed, such that no  $\omega$  or  $\beta$  phase remains in the martensite plate. The  $\beta$  and  $\omega$  shear transformation systems are parallel, which explains how the  $\beta$  phase transforms completely along with the  $\omega$  phase.

The same  $\beta$  phase deforms by  $\{332\}\langle113\rangle$  twins when present as a single-phase alloy, Ti-14.8V. Within these twins the  $\omega$  phase is present, with the same orientation relationship to the twinned region of  $\beta$  as to the parent  $\beta$  [25], which has been observed by others in  $\{332\}\langle113\rangle$  [13] and  $\{112\}\langle111\rangle$  twins [22]. In order to maintain this relationship with the twin, the  $\omega$  phase may either shear along with the  $\beta$  phase, or it may dissolve during twinning and recrystallize along its preferred directions within the twin when twinning is complete [22]. Although it is impossible to determine which of these mechanisms is operating, the result is the same, that  $\omega$  is present within the twin.

#### 7.4. Summary

Interactions between the  $\alpha$  and  $\omega$  phase are possible due to the high concentration of  $\omega$  phase within low stability  $\beta$  phases, which results in some fraction of the  $\omega$  phase in contact with the  $\alpha$  phase. The misfit strain between the  $\alpha$  and  $\omega$  phases is high, and places stress on the  $\omega$  phase along planes and directions which aid in the  $\omega \rightarrow \alpha'$  transformation. This misfit strain is eliminated by the transformation of  $\omega$  to  $\alpha'$ , which has a twin-like orientation relationship to the  $\alpha$  phase and a coherent interface. Also, a high magnitude of shear stress from slip and twinning in the  $\alpha$  phase resolves upon the  $\omega \rightarrow \alpha'$  shear transformation systems, aiding the transformation. Once the martensite plate has nucleated, it can continue growing though the beta phase due to parallel  $\omega \rightarrow \alpha'$  and  $\beta \rightarrow \alpha'$ shear transformation systems. No  $\omega$  phase remains within the stress induced martensite plate, which is in contrast to the single phase  $\beta$  alloy Ti-14.8 which deforms by twins that contained the same  $\omega$  as the parent  $\beta$  phase.

## 7.5. Conclusions

- 1. The  $\omega$  phase plays an important role in the  $\beta$  phase deformation mechanisms of two-phase titanium alloys.
- 2. Misfit strain between the  $\alpha$  and  $\omega$  phases aids in the  $\omega \rightarrow \alpha'$  transformation.
- The interfacial free energy between the α and ω phases is reduced by the ω→α' transformation. This is because the resulting stress induced martensite has a near-twin relationship and coherent interface with the α phase due to orientation relationships between the α, β, and ω phases.
- Shear stresses from slip and twinning in the α phase resolve onto the ω→α' shear systems, which aids in the ω→α' transformation, resulting in α' plates in the β phase. This is in contrast to the single-phase β alloy, where twinning is the primary deformation mechanism.

- 5. Parallel  $\omega \rightarrow \alpha'$  and  $\beta \rightarrow \alpha'$  phase transformation shear systems allow the stress induced martensite which nucleates in the  $\omega$  phase to propagate through the  $\beta$  phase.
- 6. In contrast to twins in the single phase  $\beta$  alloy Ti-14.8V, no  $\omega$  phase remains within the stress induced martensite plates in the  $\beta$  phase of Ti-8.1V.

## **Chapter 8**

## Conclusions

- 1. The tensile and creep deformation mechanisms of the  $\alpha$  and  $\beta$  phases of the model two-phase  $\alpha$ - $\beta$  alloys, Ti-6.0Mn and Ti-8.1V, were different from those of the component  $\alpha$  and  $\beta$  phases when present in single phase alloys with the same chemistry and microstructure.
- Both model α-β alloys with Widmanstätten microstructure deform in tension by slip and twinning in the α phase. In contrast, when the α phases with the same chemistry and similar grain size are present in single phase alloys no twinning was observed.
- 3. Ti-8.1V with Widmanstätten microstructure deforms in tension by stress induced hexagonal martensite in the β phase. This is in contrast to the single-phase β Ti-V alloy with the same chemistry, which deforms by slip and twinning. Twins in the α phase and stress induced martensite plates in the β phase were observed to coincide at the α-β interface. This is the first time that this combination of deformation products has been observed in an α-β titanium alloy.
- 4. These novel tensile deformation mechanisms were modeled in terms of  $\beta$  phase stability and interactions between the  $\alpha$  and  $\beta$  phases, including elastic interaction stresses, the  $\alpha$  phase acting as a template for hexagonal martensite nucleation, and shear stresses from  $\alpha$  and  $\beta$  phase deformation products.
- 5. The creep deformation mechanisms of Ti-6.0Mn and Ti-8.1V with Widmanstätten microstructures were similar to the tensile deformation mechanisms, including

slip and twinning in the  $\alpha$  phase of both alloys, and the formation of stress induced hexagonal martensite in the  $\beta$  phase of Ti-8.1V. These novel creep deformation mechanisms were also modeled in terms of interactions between phases.

- 6. This is the first time that stress induced martensite has been reported as a creep deformation mechanism in an  $\alpha$ - $\beta$  titanium alloy.
- Several mechanisms for time-dependent growth of martensite were proposed, including growth controlled by slip or time dependent twinning in the α phase or an inherent time-dependent mechanism.
- 8. Interphase interface sliding occurs in several Ti-Mn alloys but not Ti-8.1V. Several contributions to this difference in sliding are discussed, including the stability of the β phases, differences in the α to β strength ratio, and differences in the α-β interface structure. A model based on locking / unlocking of growth ledges is proposed to explain anisotropic interphase interface sliding.
- 9. The tensile deformation mechanisms of Ti-8.1V with an *equiaxed* microstructure were similar to those of Ti-8.1V with a *Widmanstätten* microstructure. A Burgers orientation relationship was confirmed between the  $\alpha$  and  $\beta$  phases in the equiaxed microstructure, hence the model for interactions between phases proposed for *Widmanstätten* alloys can be used to explain these deformation mechanisms.
- 10. No extended chains of  $\alpha$  and  $\beta$  phase deformation products were observed to in the equiaxed alloy due to the random distribution of  $\alpha$  grain orientations. These chains were observed in Ti-8.1V with a Widmanstätten microstructure. This

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difference contributes to the higher yield stress of the alloy with equiaxed microstructure.

11. Interactions between the  $\alpha$  and  $\omega$  phases can affect the  $\beta$  phase deformation mechanisms of a two-phase  $\alpha$ - $\beta$  alloy. Strain placed on the  $\omega$  phase at the  $\alpha$ - $\omega$ interface aids in the  $\omega \rightarrow \alpha'$  transformation. The  $\alpha'$  that results from the transformation has a lower misfit strain with the  $\alpha$  phase compared to the  $\alpha$ - $\omega$ interface. Additionally, the resolved shear stresses from slip and twinning in the  $\alpha$ phase acts on the shear systems of the  $\omega \rightarrow \alpha'$  transformation.

### Chapter 9

## **Suggestions for Future Work**

- 1. Tensile and creep testing of equiaxed Ti-8.1V alloys at slightly elevated temperatures 358-458 K. These studies will be useful for studying the tensile and creep deformation mechanisms over a larger temperature range, but may also give clues to the extent of the alteration of the  $M_s$  temperature of the  $\beta$  phase due to interactions with the  $\alpha$  phase. It is expected that as the testing temperature is raised that the  $\beta$  phase deformation mechanism will change from stress induced martensite to twinning. The activation energy can also be calculated for the creep deformation processes, and correlated with published values of activation energy for stress induced martensite and TEM observations.
- During the course of the present work several mechanisms of time dependent martensite growth were proposed for creep deformed Ti-8.1V. Interrupt creep testing of Ti-8.1V should be performed in order to investigate the growth rate of stress induced martensite. Time dependent twinning was observed in single phase α and β alloys using this method, and time dependent martensite growth is an exciting possibility that deserves further study.
- If the stress induced martensite in Ti-8.1V is time dependent, then additions of oxygen should make the growth of martensite more difficult due to the need to diffuse into or out of interstitial sites in the martensite across the β-α' interface. Therefore, the effect on the creep deformation rate of α-β titanium alloys with varying oxygen concentrations could be studied. Since oxygen is an α stabilizer,

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additional V should be added to maintain the same  $\beta$  phase stability, as it is already known that the stability of the  $\beta$  phase has an effect on the creep deformation mechanisms.

- 4. Classification of specific deformation features observed in optical and SEM micrographs by analytical SEM techniques such as electron backscatter diffraction (EBSD), or TEM sample preparation by focused ion beam. There will always be an element of uncertainty to whether the local deformation products observed in TEM are the same types of deformation products observed on a larger scale. Using these techniques in conjunction with TEM would remove all doubt.
- 5. FEM modeling or other simulation to model and predict the creep deformation behavior of two-phase alloys incorporating the results of this study. Previous attempts to predict the creep deformation behavior have been inadequate due to a lack of consideration for interactions between phases.
- 6. Tensile tests should be conducted beyond 3% strain to fracture in order to study the effect of the newly discovered deformation mechanisms in Ti-8.1V on fracture mechanics. In this regard, fatigue and creep-fatigue testing could also be performed.
- 7. It was shown during the course of this investigation that the nanoscale  $\omega$  phase can be completely transformed to hexagonal martensite during tensile and creep deformation. Attempts could be made to transform this phase into  $\alpha'$  without changing the particle size or transforming the  $\beta$  phase, resulting in a nanoscale martensite/ $\beta$  composite. This might be accomplished by thermomechanical treatment, and would result in an extremely tough  $\alpha$ - $\beta$  alloy.

# Appendix A



# **Ti-Mn and Ti-V Phase Diagrams and Alloy Compositions**

Phase diagrams of the solute rich ends of the Ti-Mn [63] and Ti-V [62] systems. Alloy compositions are indicated on the diagram, and listed below.

Alloy	Alloy #	Microstructure	α phase	β Phase
			composition	composition
Ti-0.4wt%Mn	1	~100% a	Ti-0.4wt%Mn	N/A
Ti-6.0wt%Mn	2	~46% a, ~54% ß	~Ti-0.4wt%Mn	~Ti-13.0wt%Mn
Ti-13.0wt%Mn	3	~100% ß	N/A	Ti-13.0wt%Mn
Ti-1.5wt%V	4	~100% a	Ti-1.6wt%V	N/A
Ti-8.1wt%V	5	~51% a, ~49% β	~Ti-1.6wt%V	~Ti-14.8wt%V
Ti-14.8wt%V	6	~100% β	N/A	Ti-14.8wt%V

# **Appendix B**

# **Tensile and Creep Specimen Specifications**

Specimen with flats for tensile and creep testing (to be cut by electric discharge machining) for pre and post testing optical and SEM microscopy [27].



Method for calculating the cross sectional area of the gage length for specimens with the above geometry.



Cross section of tensile or creep specimen

$$Area = \frac{db}{2}\cos\left(\frac{\theta}{2}\right) + \frac{\theta}{180}\pi\left(\frac{d}{2}\right)^2$$
$$\theta = 2\sin^{-1}\left(\frac{b}{d}\right)$$

# Appendix C

# **Titanium Etch Solutions**

A etch is used to etch grain boundaries. Etching times of 10-20 seconds should be used. Only a small amount of A- etch should be prepared at one time. Do not store A etch or A etch waste. DES should be called immediately for waste pickup.

A Etch	
Chemical	Amount
Hydrofluoric Acid - HF 50%	2.5 ml
Nitric Acid - HNO <sub>3</sub>	2.5 ml
Glycerine	5.0 ml

R etch is a color etchant which dyes the  $\alpha$  phase The  $\alpha$  phase will then appear black optically, but with be the lighter phase using SEM. Etching times of 10-20 seconds should be used. R etch can be stored in a sealed polyethylene bottle.

R Etch		
Chemical	Amount	
Benzalkonium Chloride	18.5 g	
Ethanol	35 ml	
Glycerine	40 ml	
Hydrofluoric Acid – HF 50%	25 ml	

Dilute titanium etch is used prior to depositing gold fiducial grid lines in order to facilitate the removal of photoresist.

Dilute Ti Etch		
Chemical	Amount	
Water	194 ml	
Nitric Acid – HNO <sub>3</sub>	4 ml	
Hydrofluoric Acid – HF 50% 2 ml		

# **Appendix D**

# **Calculated Dimple Depths for South Bay Dimplers**

Chem Nuc 1119 Wheel radius (mm) 13.284 J.M Patterson Wheel Radius (mm) 15.91

Measuring Microscope J.M Patterson 61 gradients at 2x = 3.02 mm 1 grad = (mm) 0.049508197

Dimple Dia (grad, 2x)	Dimple dia (mm)	Depth CN (um)	Depth J.M.P (um)
2	0.10	0.09	0.08
4	0.20	0.37	0.31
6	0.30	0.83	0.69
8	0.40	1.48	1.23
10	0.50	2.31	1.93
12	0.59	3.32	2.77
14	0.69	4.52	3.77
16	0.79	5.91	4.93
18	0.89	7.47	6.24
20	0.99	9.23	7.70
22	1.09	11.17	9.32
24	1.19	13.29	11.10
26	1.29	15.60	13.02
28	1.39	18.09	15.10
30	1.49	20.77	17.34
32	1.58	23.64	19.73
34	1.68	26.69	22.28
36	1.78	29.92	24.98
38	1.88	33.35	27.83
40	1.98	36.95	30.84
42	2.08	40.75	34.01
44	2.18	44.73	37.33
46	2.28	48.89	40.80
48	2.38	53.25	44.43
50	2.48	57.79	48.22
52	2.57	62.51	52.16
54	2.67	67.43	56.25
56	2.77	72.53	60.51
58	2.87	77.82	64.91
60	2.97	83.29	69.48

## **Appendix E**

## JEOL JEM-4000FX Operation and Trouble Shooting Guide

The JEOL JEM-4000FX is a complex and highly tuned scientific instrument. The complete details of its operation and specialized techniques that can be used are beyond the scope of this document, but below are critical procedures and precautions that must be followed in order to insure the safety of the instrument. Additionally, I have included the start-up procedure, as power outages due to weather and building renovation are common.

#### **Special Precautions**

- When removing the specimen holder, make sure that the load-lock chamber is allowed to vent before completely removing the holder. If not, the microscope will continue pumping on atmosphere.
- 2. When loading the specimens into the specimen holder, check the spring fasteners under the optical microscope to make sure that your specimens are secure. A specimen falling into the column and down onto the screen/camera would be disastrous. Also, check the o-ring under the microscope for splits and debris such as lint. Remove debris *carefully* with forceps.
- 3. When inserting specimen holder into the microscope, turn to the right until the specimen holder drops one level and STOP. Allow the load lock to pump down until the red indicator light on the load-lock comes on. This takes ~15 minutes.

- 4. Only open the load lock gate valves that separate the column from the gun and camera chambers after the specimen has been inserted into the column and the red load lock light remains on. After opening the orange load lock button on the panel, you can then turn on the gun and lens currents in the left drawer.
- 5. Turn on the filament voltage first by pressing the red HT button. After the current readout automatically increases to ~91, slowly turn the gun current knob to position 1. Hold at this position and each subsequent position to position 4 for 2 minutes each. Then increase the current knob to 4.5, and allow the filament to warm up for 15 minutes. After this time you can increase the filament current until the stop (readout will be ~100) and use the microscope.
- 6. When shutting down the microscope, first turn down the filament current, then turn off the filament voltage. Next, close the column/gun load lock gate valves and turn off the lens and gun power. Remove the specimen holder by first zeroing all tilts.
- 7. The room air conditioning must be running in order for the microscope to operate properly. The room thermostat should be set year round to 70°F and "cool".

#### **Restart procedure:**

The JEOL microscope will shut down and/or fail to restart for 2 primary reasons: (1) The column or gun pressure is too high or (2) the ion pump and voltage generator are insufficiently cooled. These are usually caused by power outages, but have also been triggered by problems with the water chiller and rooftop fan.

If the TEM is shut down, first check the side panel. A red LED most likely will indicate that the cooling water is insufficient. Remove the TEM panel and press the red "Reset" button. Next, check the water chiller that is located in the hallway under the stairs. Start the water cooler by holding the start switch up. In most instances of a power failure this will be sufficient to restart the chiller. If this does not work, reset the high pressure shutoff breakers behind the front chiller panel. Start the TEM by turning the key on the panel to the right until the pumps begin.

Check to make sure that the gauges located in the right rear corner of the TEM room are turned on. If the red indicator lights are off, turn the gauge switches to the "off" position and then "on". If these gauges are not reset the TEM will sense that the column and gun chamber pressures are high, and the microscope will not operate.

## Appendix F

## **JEOL JEM-4000FX Diffraction Pattern Rotation Calibration**

The image in a TEM rotates to various degrees depending on the magnification. In contrast, the diffraction pattern will remain at the same rotation regardless of which magnification or selected area aperture is used. In order to properly orient the diffraction pattern and the image, film recording selected area diffraction patterns must be rotated with respect to the image film. This calibration is essential for labeling crystallographic directions and identifying dislocation types. Rotation calibration for the JEOL JEM-4000FX was performed using a cross-sectional TEM specimen of SiC epitaxial film on (100) Si provided by Prof. Salamanca-Riba. Below are the rotation angles of the diffraction pattern for common magnification settings. Rotation for magnifications between those measured can be extrapolated.

Magnification (x1000)	Rotation (°CCW)
20	7.5
30	8
40	4
50	3.5
80	3.5
120	3.5
150	3.5
200	61
300	63
400	63

Film must be viewed in this orientation prior to rotating.

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## Appendix G

# $\{10\overline{1}2\}_{\alpha}$ and $\{10\overline{1}1\}_{\alpha}$ Twin Identification

In previous studies  $\{10\overline{1}2\}_{\alpha}$  twins were misidentified as  $\{10\overline{1}1\}_{\alpha}$  twins. In order to prevent future misidentification the author has prepared the following short treatment of twin identification in  $\alpha$  titanium.

In order to identify a twin by selected area diffraction, the TEM specimen must be tilted such that both the twinning plane normal and the twinning direction are perpendicular to the beam direction. Below are the matrix zone axes for identification and degree of rotations for common  $\alpha$  phase twins [18, 19]. The degree of rotation will vary slightly with changes in the c/a ratio. Notice that for any given twin only one of the three independent  $\langle 1\overline{2}10 \rangle$  or  $\langle 1\overline{1}00 \rangle$  zone axes will allow for twin identification.

Twin	Zone axis	<b>Rotation angle °</b>
$\left<10\overline{1}1\right>\overline{1}012$	$\langle 1\overline{2}10 \rangle$	~122
$\left< 10\overline{1}2 \right> \overline{1}011 \right>$	$\langle 1\overline{2}10 \rangle$	~94
$\left\{11\overline{2}2\right\}1/3\left(11\overline{2}\overline{3}\right)$	$\left< 1\overline{1}00 \right>$	~63
$\left\{11\overline{2}1\right\}1/3\left\langle\overline{1}\ \overline{1}\ 26\right\rangle$	$\left<\overline{1}100\right>$	~34

Below are schematics of the hcp structure of the  $\alpha$  phase, shown from the  $[0001]_{\alpha}$  perspective and  $[1\overline{2}10]_{\alpha}$  perspective. Indicated are traces of the  $(10\overline{1}1)_{\alpha}$  and  $(10\overline{1}2)_{\alpha}$  planes.





When  $\{10\overline{1}1\}_{\alpha}$  or  $\{10\overline{1}2\}_{\alpha}$  twinning occurs atoms in the twinned structure and the matrix will be mirrored over the twin plane. This orientation can be represented schematically as a rotation of the lattice around a pole perpendicular to both the twin plane normal and the twinning direction (which is the same as the zone axis required for identification by selected area diffraction discussed above). Below the twinned crystal lattice is drawn in relation to the matrix. The twinned crystal structure is drawn in red.



In order to identify a twin selected area diffraction patterns must be taken from, at a minimum, the matrix and the twin/matrix interface. It is also preferable to take the diffraction pattern of the twinned region if possible. The selected area diffraction patterns taken from the twin/matrix interface along the correct zone axis for a particular  $\{10\overline{1}1\}_{\alpha}$  or  $\{10\overline{1}2\}_{\alpha}$  twin will be similar to the examples shown below. The contribution of reflections from the matrix and twin are illustrated at right.

# $\{10\overline{1}2\}_{\alpha}$ twin/matrix interface selected area diffraction pattern



 $\{10\overline{1}1\}_{\alpha}$  twin/matrix interface selected area diffraction pattern



## **Appendix H**

## $\alpha$ and $\omega$ Phase Coordinate Conversion to Parallel Directions in $\beta$

#### 4 to 3 Coordinate Conversion Equations

Conversion of 4 coordinate directions [hklq] for the  $\alpha$  phase (hcp) or  $\omega$  phase (P6/mmm) to directions in the 3 coordinate [xyz] Cartesian system. Consideration for the c/a ratio hexagonal crystal must be taken to calculate the correct z coordinate with respect to the x and y coordinates.



#### $\alpha \rightarrow \beta$ Coordinate Conversion Matrix

Conversion matrix for the calculation of parallel directions in the  $\beta$  phase to a given direction in the  $\alpha$  phase of a two-phase titanium alloy with a Burgers orientation relationship  $\langle 1\overline{2}10 \rangle (0001)_{\alpha} // \langle 1\overline{1}1 \rangle (110)_{\beta}$ . If this conversion is used for planes, care must be taken to make sure that the plane normal is actually perpendicular to the plane. Planes and directions in the HCP system are not necessarily perpendicular as they are in a cubic system.

$$\begin{bmatrix} -.2638797 & -.81902 & \frac{\sqrt{2}}{2} \\ .2638797 & .81902 & \frac{\sqrt{2}}{2} \\ -1.8392 & -1.72853 & 0 \end{bmatrix}$$
### $\omega$ to $\beta$ Phase Parallel Direction Transformation Matrices

ω - Orientation 1 -	$ \begin{bmatrix} 0001 \end{bmatrix}_{\omega} / / \begin{bmatrix} 1 \overline{1} 1 \end{bmatrix}_{\beta} \\ \begin{bmatrix} \overline{1} 010 \end{bmatrix}_{\omega} / / \begin{bmatrix} \overline{1} 12 \end{bmatrix}_{\beta} \\ \begin{bmatrix} \overline{1} 2 \overline{1} 0 \end{bmatrix}_{\omega} / / \begin{bmatrix} 110 \end{bmatrix}_{\beta} $	$\begin{bmatrix} 0\\ -\frac{1}{\sqrt{2}}\\ -\frac{1}{\sqrt{2}} \end{bmatrix}$	$\frac{\frac{2}{\sqrt{6}}}{\frac{1}{\sqrt{6}}}$ $-\frac{1}{\sqrt{6}}$	$\frac{1}{\sqrt{3}} \\ -\frac{1}{\sqrt{3}} \\ \frac{1}{\sqrt{3}} \\ \frac{1}{\sqrt{3}} \end{bmatrix}$
ω - Orientation 2 -	$ \begin{bmatrix} 0001 \end{bmatrix}_{\omega} / / \begin{bmatrix} \overline{1} & 11 \end{bmatrix}_{\beta} \\ \begin{bmatrix} \overline{1} & 010 \end{bmatrix}_{\omega} / / \begin{bmatrix} 1 & \overline{1} & 2 \end{bmatrix}_{\beta} \\ \begin{bmatrix} 1\overline{2} & 10 \end{bmatrix}_{\omega} / / \begin{bmatrix} 110 \end{bmatrix}_{\beta} $	$\begin{bmatrix} 0\\ \frac{1}{\sqrt{2}}\\ -\frac{1}{\sqrt{2}} \end{bmatrix}$	$-\frac{2}{\sqrt{6}}$ $-\frac{1}{\sqrt{6}}$ $-\frac{1}{\sqrt{6}}$	$-\frac{1}{\sqrt{3}}$ $\frac{1}{\sqrt{3}}$ $\frac{1}{\sqrt{3}}$ $\frac{1}{\sqrt{3}}$
ω - Orientation 3 -	$\begin{bmatrix} 0001 \end{bmatrix}_{\omega} / / \begin{bmatrix} 111 \end{bmatrix}_{\beta} \\ \begin{bmatrix} \overline{1} & 010 \end{bmatrix}_{\omega} / / \begin{bmatrix} \overline{1} & \overline{1} & 2 \end{bmatrix}_{\beta} \\ \begin{bmatrix} \overline{1} & 2 & \overline{1} & 0 \end{bmatrix}_{\omega} / / \begin{bmatrix} \overline{1} & 10 \end{bmatrix}_{\beta}$	$\begin{bmatrix} \frac{1}{\sqrt{2}} \\ 0 \\ -\frac{1}{\sqrt{2}} \end{bmatrix}$	$-\frac{1}{\sqrt{6}}$ $\frac{2}{\sqrt{6}}$ $-\frac{1}{\sqrt{6}}$	$\frac{1}{\sqrt{3}} \\ \frac{1}{\sqrt{3}} \\ \frac{1}{\sqrt{3}} \\ \frac{1}{\sqrt{3}} \end{bmatrix}$
ω - Orientation 4 -	$\begin{bmatrix} 0001 \end{bmatrix}_{\omega} / / \begin{bmatrix} \overline{1} & \overline{1} & 1 \end{bmatrix}_{\beta} \\ \begin{bmatrix} \overline{1} & 010 \end{bmatrix}_{\omega} / / \begin{bmatrix} 112 \end{bmatrix}_{\beta} \\ \begin{bmatrix} 1\overline{2} & 10 \end{bmatrix}_{\omega} / / \begin{bmatrix} \overline{1} & 10 \end{bmatrix}_{\beta}$	$\begin{bmatrix} -\frac{1}{\sqrt{2}} \\ 0 \\ -\frac{1}{\sqrt{2}} \end{bmatrix}$	$\frac{1}{\sqrt{6}} \\ -\frac{2}{\sqrt{6}} \\ -\frac{1}{\sqrt{6}}$	$-\frac{1}{\sqrt{3}}\\-\frac{1}{\sqrt{3}}\\\frac{1}{\sqrt{3}}\\\frac{1}{\sqrt{3}}\end{bmatrix}$

Conversion of important alpha phase 4 coordinate directions to 3 coordinate directions and parallel beta phase vectors

### Directions

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2.59808	-2.5981	0	-0.866	-0.866	-1.7321
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3.2	-0.866	1.5
3.2	-1.7321	0
3.2	1.73205	0
3.2	-0.866	-2
3.2	0.86603	1.5
0	-1.7321	0
0	-0.866	-2

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0	1.73205	3.2
0	-1.7321	3.2
1.5	-0.866	3.2
-2	0.86603	3.2
1.5	0.86603	1.6
-2	-0.866	1.6

1.6	1.6	1.6	1.6	1.6	
-0.866	1.73205	-1.7321	-0.866	0.86603	
-2	0	0	1.5	-2	

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-2	1.5

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0.26	1.732	-1.99

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-1.241 0.12979

1.241

1.47248

0.7504

-0.75

Parallel Beta Phase Direction

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0.707 0.7071

-1.4725	0.381	1.882
-0.1298	3.5036	1.021
0.12979	1.0212	3.504
1.60226	1.7716	2.753
-1.6023	2.7532	1.772
1.47248	3.0128	1.512
-1.4725	1.512	3.013
1.60226	-0.491	0.491

-0.1298	2.3726	-0.11
0.12979	-0.11	2.373
1.60226	0.6405	1.622

3.33431

-2.223

2.223

0.381 1.8818 1.47248

-1.6023

1.6222

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### Important $\alpha$ and $\omega$ Directions and Planes Converted

**Appendix I** 

### Conversion of important alpha phase 4 coordinate planes to 3 coordinate planes and parallel beta phase vectors

0.938	0.938	0.938	0.938	0.938	0.938
-0.866	0.86603	-1.7321	1.73205	0.86603	-0.866
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1.73205 0.86603 -0.866

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Parallel E	-0.09	1.413
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ase Plan	1.47248	-1.4725	1.60226	-1.6023	-0.1298	0.12979	
Beta Ph	1.4134	-0.087	0.1722	1.1538	1.9042	-0.578	
Parallel	-0.09	1.413	1.154	0.172	-0.58	1.904	

1.47248	-1.4725	1.60226	-1.6023	-0.1298	0.12979	
2.0762	0.5754	0.835	1.8167	2.567	0.0846	
0.575	2.076	1.817	0.835	0.085	2.567	

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// beta direction - omega 2	-0.6 0.5774	
omega 1	.57735	

// beta direction -0.577 -0.577

101 O	101.0-	1 4142	7
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-0.71 0.7071 1.41421

-1.414 -0.7071

-0.707 0.7071

-1.41 -0.71

-1 0 -1

0.7071	1.4142	2.1213	-2.121	-3E-13
- -	0.71	0	-2.1	2.12

0 -2.121 -2.1213 2.121 2.1213 3.2E-13

0 0 0

3E-13 2.12132

-2.12

// beta direction - omega 4

// beta direction - omega 3

0.577 0.5774 0.57735

-0.71 -0.707 1.41421

-0.707 -0.7071

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-1.414 0.7071

0.707

	0.7071	1.4142	0.7071	0
5	0.71	-0.7	-1.4	-2.1

0	-2.121	2.1213
-2.1	2.12	0

ectors of t	twinning	g she	ear sy	1 Parallel plane 1.241 -1.2 ystems	-1 0 0] e in beta 241 0.13	-1 0 1 0 Parallel plane in beta -0.75 0.75 1.472	0 -1 1 0 Parallel plane in beta 0.491 -0.49 1.6
lane	Dire	ection	c				
3 3 -2	-	-	e	0.056		-0.36	-0.305
3 -3 2	7	-	e	-0.772		0.936	0.163
-3 3 2	~	÷	e	0.477		0.523	-
3 3 2	-	÷	ကု	0.056		-0.36	-0.305
3-23	-	ო	-	-0.06		0.473	0.413
3 2 -3	7	ო	-	-0.582		0.134	-0.448
3 2 3	ſ	ကု	-	-0.578		0.25	-0.328
	ŀ					1	

Twins
Beta
b
Stress
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Resolved
Alpha -
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Slip
Prism

In beta

<sup>D</sup>arallel direction 1.992

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Parallel direction in bet

Parallel direction in beta

Alpha slip direction

32 -1.7

I.732

-3.07

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Slip plane in alpha

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Slip plane in alpha

Alpha slip direction

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-1.992

Slip plane in alpha

Alpha slip direction

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י. י	<u>د</u> م	0.477	C7C.0	_
3 3	2 1 1 -3	0.056	-0.36	-0.305
3 -2	3 1 3 1	-0.06	0.473	0.413
32-	3 -1 3 1	-0.582	0.134	-0.448
3 2	3 1 -3 1	-0.578	0.25	-0.328
-3 2	3 1 3 -1	0.149	-0.5	-0.353
-2 3	3 3 1 1	-0.149	0.502	0.353
23	3 -3 1 1	0.582	-0.13	0.448
23-	3 3 -1 1	0.578	-0.25	0.328
2 -3	3 3 1 -1	0.06	-0.47	-0.413

### Appendix J

Prism Slip in Alpha - Resolved Shear Stress on Martensite Shear System I

1	0				0		
on	-2	in beta	-3.07	าล	0	beta	0.13
ip directi	1	direction	-0.26	he in alph	-	plane in	-1.241
Alpha sl	-	Parallel	0.26	Slip plar	-	Parallel	1.241

Parallel direction in beta -1.992 1.992 1.3

Slip plane in alpha

Parallel plane in beta

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0.491 -0.49

C

Alpha slip direction

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Vectors of martensite shear system I

	-0.372	0.492	0.93	-0.372	0.246	-0.515	-0.142	-0.465	0.465	0.515	0.142	-0.246
	-0.44	1	0.783	-0.44	0.5	-0.06	0.383	-0.39	0.392	0.058	-0.38	-0.5
	0.069	-0.508	0.147	0.069	-0.254	-0.456	-0.525	-0.073	0.073	0.456	0.525	0.254
Direction	2 1 1 -1	2 1 -1 1	2 -1 1 1	2 1 1 1	1 -1 1	1 1 -1	1 1 1 1	1 -1 1 1	1 -1 1 1	1 1 1 1	1 1 -1	1 -1 1
Plane	1 1 2	-1 1 2	1 -1	1 1 -2	1 2	-1 2	1 -2 、	1 2 -	2 1	-2 1 、	2 -1 、	2 1 -

Prism Slip in Alpha - Resolved Shear Stress on Martensite Shear System II

	0			ĺ	0		
ection	1 -2	ion in beta	26 -3.07	alpha	-1 0	in beta	41 0.13
ip dire		direct	°.	ne in a		plane	-1.2
Alpha sl	1	Parallel	0.26	Slip plar	1	Parallel	1.241

	0	_ ا			0		
Ipha slip direction	1 -2 1	arallel direction in beta	1.732 -1.732 1.732	lip plane in alpha	-1 0 1	arallel plane in beta	-0.75 0.75 1.472
d₽	-	oal	<del>.</del> -	Slip		Jai	Ŷ

Parallel direction in beta

3

-1.992 1.992

Slip plane in alpha

Parallel plane in beta

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0.491 -0.49

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Alpha slip direction

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Vectors of martensite shear system II

	-0.133	0.805	0.426	0.512	0	0	-0.379	0.379	-0.512	0.426	0.805	0.133
	-0.48	0.678	0.866	0.289	0	0	0.188	-0.19	-0.29	0.866	0.678	0.476
							)					)
	0.343	0.127	-0.44	0.224	0	0	-0.567	0.567	0.224	-0.44	0.127	-0.343
							-		-			-
on	-	-	-	-	-	-	-	-	÷	-	-	٢
ecti	-	-	7	-	7	-	-	-	-	7	-	-
Dir	1	7	-	-	-	7	1	-	-	-	7	1
	1	-	-	-	0	0	0	0	-	-	-	1
Ð	0	0	0	0	-	-	-	-	-	-	-	-
Plan	١	Ļ	7	ŗ	Ļ	-	-	Ļ	0	0	0	0

### Basal Slip in Alpha - Resolved Shear Stress on Beta Twins

	0			ĺ	-		
	-2	beta	3.07		0	ta	0
direction	1	ection in	-0.26 -	n alpha	0	ine in be	<b>.</b>
lpha slip (	1	arallel dir	0.26	lip plane i	0	arallel pla	Ļ
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	0				-		
Alpha slip direction	1 -2 1	Parallel direction in beta	1.732 -1.732 1.732	Slip plane in alpha	0 0 0	Parallel plane in beta	1 1 0

Parallel direction in beta -1.992 1.992 1.3

Slip plane in alpha

Alpha slip direction

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Parallel plane in beta

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Vectors of twinning shear system

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Dir	
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Pla	

0.274	0	0	-0.274	-0.268	0.503	-0.542	0.777	0.777	0.503	-0.542	-0.268
0.367	0	0	-0.37	0.814	-0.5	0.446	-0.13	-0.13	-0.5	0.446	0.814
.632	0	0	.632	.518	.023	.114	.655	.655	.023	.114	.518
- 0-			0	Ŷ	° P	Ö	° P	° P	° P	Ö	° P
1 3	1 3	-1 3	1 -3	3 1	3 1	-3 1	3 -1	1 1	1 1	-1 1	1 -1
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-2	2	2	2	3	Ϋ́	Э	З	Э	З	Ϋ́	З
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3	ო	ကု	3	с	с	ო	ကု	Ņ	2	2	2

## Basal Slip in Alpha - Resolved Shear Stress on Martensite Shear System I

	0				-		
irection	1 -2	ction in beta	0.26 -3.07	ı alpha	0 0	ne in beta	1 0
Alpha slip d	٢	Parallel dire	0.26 -	Slip plane ir	0	Parallel plar	٢

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n		in t	1.73	a		oeta	
∋ctic	2	ion	N	lph	0	in	<b>.</b>
dire		rect	.73	in a		ane	
slip		ldi	7	ane		l pl	
na s	-	alle	732	pla	0	alle	<b>`</b>
٩lp		Jan	-	Slip		Jan	

Parallel direction in beta -1.992 1.992 1.3 Slip plane in alpha

Parallel plane in beta

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Alpha slip direction

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	0.192	0	0	-0.19	0.866	-0.48	0.674	-0.29	-0.29	-0.48	0.674	0.866
	-0.331	0	0	0.331	-0.413	-0.249	-0.082	-0.58	-0.58	-0.249	-0.082	-0.413
Direction	1 1 -1	1 -1 1	-1 1 1	1 1 1	1 -1 1	1 1 -1	1 1 1	-1 1 1	-1 1 1	1 1 1	1 1 -1	1 -1 1
Plane	1 1 2	-1 1 2	1 -1 2	1 1 -2	1 2 1	-121	1 -2 1	12-1	2 1 1	-2 1 1	2 -1 1	2 1 -1

-0.423 0.71 -0.567 0.853 0.853 0.853 0.71 -0.567

-0.423

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Alpha slip direction	1 1 -2	Parallel direction in beta	0.26 -0.26 -3.07	Slip plane in alpha	0 0 0	Parallel plane in beta	1 1 0

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oha		ral	.73	p pl	)	Irall	
AI		0	-	SII		0	

Parallel direction in beta -1.992 1.992 1.3 Slip plane in alpha

Alpha slip direction

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Parallel plane in beta

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Vectors of martensite shear system II

	-0.24	0.493	0.244	0.493	-0.485	0.985	0.737	0.737	0.493	-0.24	-0.493	-0.244
	5	2	5	2	1	3	2	2	7	5	2	5
	0	-0.1	·0-	-0.1		-0.3	9.0-	9.0-	-0.1	0	0.16	0
	8	2	8	2	0	2	2	2	0	8	2	8
	-0.23	iee.0-	0.23	i88.0-	-0.47(	-0.6	.60'0-	.60'0-	-0.33!	-0.23	82.0	-0.23
Direction	1 1 -1	-1 1 1	1 -1 1	1 1 1	1 -1 1	-1 1 1	1 1 -1	1 1 1	1 1 -1	1 -1 1	-1 1 1	1 1 1
	-	-	-	-	0	0	0	0	-	-	-	-
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Alpha twinning direction 1 0 -1 Parallel direction in beta 3.013 1.512 -1.47 Shear plane in alpha -1 0 1 Parallel plane in beta -0.087 1.4134 1.472

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Alpha twinning direction	-1 0 1 2	parallel direction in beta	1.512 3.013 1.472	Shear plane in alpha	1 0 -1 1	arallel plane in beta	1.413 -0.087 -1.47
Alph		Para	1.5	Shea		Para	1.4

Parallel direction in beta 1.772 2.753 -1.6 Shear plane in alpha

Parallel plane in beta

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0

1.154 0.172 1.6

0.914 -0.419 -0.027 0.275

Alpha twinning direction

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Vectors of twinning shear direction

	0.248	0.158	-0.37	0.813	-0.01	0.488	0.044	-0.55
	13	58	872	48	46	17	999	34
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0.449	0.091	-0.5	0.172	0.169	-0.388	0.706	0.396
1.01	488	044	).55	).34	).82	366	).15
Υ 	0	0		φ 		0.	-
0.146	0.817	-0.366	0.34	0.553	-0.488	-0.044	0.008
-	1	1	-1	1	1	1	-1
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1011 Twinning in Alpha - R	esolved Shear Stress on	n Beta T	wins (continuec	-
	Alpha twinning direction		Alpha twinning	directior
	0 -1 1	2	t- -	0
	Parallel direction in beta	1	Parallel directio	n in beta
	2.753 1.7716 1.602		3.504 1.021	0.13
	Shear plane in alpha		Shear plane in a	alpha

Alpha twinning direction	-1 1 0 2	Parallel direction in beta	1.021 3.504 -0.1	Shear plane in alpha	1 -1 0 1	Parallel plane in beta	1.904 -0.58 0.1	
Alpha twinning direction	1 -1 0 2	Parallel direction in beta	3.504 1.021 0.13	Shear plane in alpha	-1 1 1 0 1	Parallel plane in beta	-0.58 1.904 -0.13	

-1.6

1.1538

0.172

Parallel plane in beta

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0.345	0.285	0.364	0.294		0.719	0.02	-0.098	-0.666	0.083	-0.655	0.725	-0.041
0.294	0.285	0.364	0.345		0.041	0.655	-0.73	-0.08	0.666	-0.02	0.098	-0.72
						1						(
0.275	-0.419	-0.027	0.914		-0.396	0.388	-0.706	-0.169	-0.172	-0.091	3.0	-0.449
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Alpha twinning direction	1 0 -1	Parallel direction in beta	3.013 1.512 -1.47	Shear plane in alpha	-1 0 1	Parallel plane in beta	-0.087 1.4134 1.472

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pha twinning direction -1 0 1	arallel direction in bet	 1 0 1	arallel plane in beta	.413 -0.087 -1.47

Parallel direction in beta 1.772 2.753 -1.6

Shear plane in alpha

Parallel plane in beta

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1.154 0.172

Alpha twinning direction

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Vectors of martensite shear system I

Direction
Plane

	0.894	-0.375	-0.17	0.559	0.217	0.139	-0.28	0.182	0.012	-0.279	0.755	0.592
	0.501	0.008	-0.27	0.796	0.008	0.232	0.036	-0.31	-0.58	-0.83	0.268	3E-04
	0.796	0.008	-0.269	0.501	-3E-04	0.832	-0.268	0.582	0.313	-0.232	-0.036	-0.008
5	-1	1	١	١	1	-1	1	1	1	1	-1	1
5	1	7	-	-	7	-	-	-	-	1	-	7
ז	1	-	7	1	1	-	1	7	7	1	-	-
	2	2	2	-2	-	-	-	-	-	1	-	-
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1011 Twinning in Alpha - Resolved Shear Stress on Martensite Shear System I (continued)

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Alpha twinning direction	0 -1 1	Parallel direction in beta	2.753 1.7716 1.602	Shear plane in alpha	0 1 -1	Parallel plane in beta	0.172 1.1538 -1.6

	2				١		
winning direction	-1 0	I direction in beta	1.021 0.13	olane in alpha	1 0	l plane in beta	1.904 -0.13
Alpha t	ſ	Parallel	3.504	Shear p	-	Parallel	-0.58

Alpha twinning directior -1 10 2 Parallel direction in bett 1.021 3.504 -0.1 Shear plane in alpha 1 -1 0 1 Parallel plane in beta	Alpha twinning direction
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Vectors of martensite shear system I

	0.377	0.376	0.418	0.404	-0.22	0.543	-0.62	0.157	0.574	0.215	-0.17	-0.59
	0.559	-0.375	-0.17	0.894	-0.592	0.279	-0.755	-0.012	-0.182	-0.139	0.28	-0.217
	5	-	-	-	-	7	-	-	-	-	7	-
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Plan	- 1	7	-	-	-	ŗ	-	-	2	-2	2	2

0.594 -0.215 0.166 -0.574 -0.157 -0.543 0.619 0.619

0.404 0.376 0.418 0.418 0.377

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Alpha twinning direction	1 0 1	Parallel direction in beta	3.013 1.512 -1.47	Shear plane in alpha	-1 0 1	Parallel plane in beta	-0.087 1.4134 1.472

g direction	1 2	on in beta	1.472	ı alpha	-1	in beta	-1.47
oha twinning	-1 0	irallel direction	.512 3.013	iear plane in	1 0	irallel plane	.413 -0.087
A		Ъ	-	Ś		Ĕ	-

Parallel direction in beta 1.772 2.753 -1.6 Shear plane in alpha 0 -1 1 1 Parallel plane in beta 1.154 0.172 1.6

Alpha twinning direction 0 1 -1 2

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	0.444	-0.49	0.004	-0.94	0.005	-0.52	-0.02	-0.5	0.423	0.009	0.025	-0.44
	439	025	600	423	005	517	501	021	.04]	004	492	444
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ane	1 0	1 0	1 0	1 0	1	1 1	1 1	1 1	0	0 1	0 -1	0-1
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-0.091 -0.558 -0.484 -0.487 0.467 0.467 0.467 0.485 -0.355 -0.355 -0.355 -0.091 -0.091 -0.091 -0.203

0.952

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1011 Twinning in Alpha - Resolved Shear Stress on Martensite Shear System II (continued)

	2				1		
Alpha twinning direction	0 -1 1	Parallel direction in beta	2.753 1.7716 1.602	Shear plane in alpha	0 1 -1	Parallel plane in beta	0.172 1.1538 -1.6

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Alpha twinning direction -1 1 0 2 Parallel direction in beta 1.021 3.504 -0.1	Shear plane in alpha	1 -1 0 1	Parallel plane in beta	1.904 -0.58 0.1
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Vectors of martensite shear system II

Plane	ö	rection				
1 0 1	-	۲- ۲-		0.485	0.122	0.591
1 0 1	-	<u>۲</u>	1	-0.203	0.573	0.151
-1 0 1		- -	1	-0.091	0.56	0.091
-1 0 1		<b>~</b>	1	-0.596	-0.11	-0.531
1 1 0		, ,	1	-0.467	-0.47	0.468
1 1 0	-	<b>~</b>	1	-0.112	0.422	-0.422
-1 1 0	-	<b>۲</b>	1	-3E-04	0.409	-0.481
-1 1 0		<u>۲</u>	1	0.355	0.481	-0.409
0 1 1		۲ ۲	1	0.484	0.531	0.109
0 1 1		- -	1	-0.558	0.091	0.56
0 -1 1	-	1	1	-0.091	0.151	0.573
0 -1 1	-	۲	1	-0.952	-0.59	-0.122

### 1012 Twinning in Alpha - Resolved Shear Stress on Beta Twins

-	1		2		
Alpha twinning direction 1 0 -1	Parallel direction in beta	Shear plane in alpha	-1 0 1	Parallel plane in beta	0.575 2.0762 1.472

a twinning direction	-1 0 1 1	allel direction in beta	81 1.882 1.472	ar plane in alpha	1 0 -1 2	allel plane in beta	76 0.575 -1.47
Alpha t	-	Parallel	0.381	Shear p	L	Parallel	2.076

Parallel direction in beta 0.641 1.622 -1.6 Shear plane in alpha

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Parallel plane in beta 1.817 0.835 1.6

Alpha twinning direction

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Vectors of twinning shear direction

	0.662	-0.498	-0.032	0.754	-0.029	0.306	-0.733	-0.023	-0.026	-0.263	0.703	-0.093
	72	88	44	91	93	0.8	22	06	92	75	62	07
	0.6	0.1	- 0-	0.5	0'0	0	0-	0-	0.1	-0-	0.1	-0.
	0.591	0.188	-0.442	0.672	0.071	0.752	-0.162	-0.192	0.062	-0.8	0.221	-0.093
u	3	3	с	-3	-	-	٢	-1	1	1	1	-1
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a i wins (continuea)	Alpha twinning direction	1 -1 0 1	Parallel direction in beta	2.373 -0.11 0.13	Shear plane in alpha
Inning in Alpha - Resolved Shear Stress on Beta	Alpha twinning direction	0 -1 1 1	Parallel direction in beta	1.622 0.6405 1.602	Shear plane in alpha

Twins (continued)	Alnha twinning direc
- Resolved Shear Stress on Beta	Alnha twinning direction
1012 Twinning in Alpha	

Alpha twinning direction
-1 1 0 1
Parallel direction in beta
-0.11 2.373 -0.1
Shear plane in alpha
1 -1 0 2
Parallel plane in beta
2.567 0.085 0.1

2

-0.13

2.567

0.085

-1.6

Parallel plane in beta 0.835 1.8167 -1

Parallel plane in beta

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Vectors of twinning shear direction

	0.754	-0.498	-0.032
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0.339 0.377

0.384

0.339	0.434	0.384	0.403	0.448	0.535	0.403	0.488	0.356	D.444	0.501
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0.339	0.434	0.377	0.50	0.356	-0.4	-0.49	0.400	-0.45	0.535	<sup>7</sup> .0-
98	32	32	33	33	33	26	23	90	33	29
-0.49	0.0-	0.66	0.0	0.26	-0.70	0.02	0.02	-0.30	0.73	0.02
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1012 Twinning in Alpha - Resolve

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Alpha twinning direction 1 0 -1 Parallel direction in beta 1.882 0.381 -1.47 Shear plane in alpha -1 0 1 Parallel plane in beta	0.575 2.0762 1.472

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Alpha twinning direction	-1 0 1 1	Parallel direction in beta	0.381 1.882 1.472	Shear plane in alpha	1 0 -1 2	⊃arallel plane in beta	2 076 0 575 -1 47

Parallel direction in beta 0.641 1.622 -1.6 Shear plane in alpha

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Parallel plane in beta

1.6

1.817 0.835

-0.446 -0.202 0.889

0.841

-0.282 0.259 -0.65 0.087 0.087 -0.115 -0.239 0.581

0.165

Alpha twinning direction

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Vectors of martensite shear system I

Plane         Direction           1         2         1         1         0.75         0.793           -1         2         1         -1         1         0.793         0.793           1         -1         2         1         1         1         0.009         0.793           1         -1         2         1         1         1         0.793         0.793           1         2         1         1         1         0.793         0.756         0.004           -1         2         1         1         1         0.793         0.756         0.756           -1         2         1         1         1         0.793         0.756         0.767           -1         2         1         1         1         0.793         0.767         0.767           1         2         1         1         1         0.766         0.004         0.776           2         1         1         1         0.096         0.225         0.167           2         1         1         1         0.067         0.059         0.769           2         1         1													
Plane     Direction       1     2     1     1     1     0.75       -1     2     1     1     1     0.009       1     1     2     1     1     1       1     2     1     1     1     0.009       1     2     1     1     1     0.055       -1     2     1     1     1     0.056       -1     2     1     1     1     0.096       2     1     1     1     -0.225       2     1     1     1     -0.677       2     1     1     -0.677		0.793	600.0	-0.32	0.75	0.004	0.677	-0.16	0.225	-0.1	-0.59	0.116	-0.01
Plane     Direction       -1     2     1     1       -1     2     1     1     1       1     -1     2     1     1     1       1     2     1     1     1     1       1     2     1     1     1     1       1     2     1     1     1     1       1     2     1     1     1     1       2     1     1     1     1     1       2     1     1     1     1     1       2     1     1     1     1     1		0.75	0.009	-0.321	0.793	0.005	0.59	-0.116	0.096	-0.225	-0.677	0.16	-0.004
Plane     Directio       1     1     2     1     1       -1     1     2     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       2     1     1     1     1       2     1     1     1     1       2     1     1     1     1       2     1     1     1     1	c	-1	-	-	-	-	-	1	-	-	1	7	•
Plane     Dire       1     1     2     1       1     1     2     1       1     2     1     1       1     2     1     1       1     2     1     1       2     1     1     1       2     1     1     1       2     1     1     1       2     1     1     1       2     1     1     1	ctio	١	÷	~	~	÷	~	١	-	~	١	-	Ļ
Plane -1 1 2 -1 1 2 -1 1 2 -1 2 1 -1 2 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 1 -2 1 -2	Dire	-	-	ŗ	-	-	-	-	-	ŗ	-	-	Ļ
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	Plan	-	Ļ	-	-	-	Ţ	-	-	2	-2	2	~

1012 Twinning in Alpha - Resolved Shear Stress on Martensite Shear System I (continued)

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Alpha twinning direction	1-1-1	Parallel direction in beta	1.622 0.6405 1.602	Shear plane in alpha	0 1 -1	Parallel plane in beta	0.835 1.8167 -1.6

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Parallel direction in beta

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Shear plane in alpha

-0.11 2.373 -0.1

Parallel plane in beta

2.567 0.085 0.1

0.463

0.447

0.497 0.467

Alpha twinning direction

Vectors of martensite shear system I

Plane         Direction           1         2         1         1         0.489         0.467           -1         2         1         -1         0.889         0.447           1         2         1         -1         0.889         0.497           1         2         1         -1         0.846         0.497           1         1         2         1         1         0.497           1         1         -0.165         0.282         0.497           1         2         1         1         -0.165         0.282           -1         2         1         1         -0.165         0.282           -1         2         1         1         -0.202         0.497           1         2         1         1         -0.165         0.282           -1         2         1         1         -0.261         0.196           2         1         1         1         -0.265         0.326           2         1         1         1         0.65         0.326           2         1         1         1         0.282         0.326 <th></th>													
Plane         Direction           1         2         1         1         1         0.889           -1         2         1         -1         0.889            -1         2         1         -1         1         0.889           1         1         2         1         -1         1         0.841           1         1         2         1         1         1         -0.202           1         2         1         1         1         -0.202           -1         2         1         1         -0.165            -1         2         1         1         1         -0.165           -1         2         1         1         1         -0.165           2         1         1         1         -0.087           2         1         1         1         -0.259           2         1         1         1         1         -0.269           2         1         1         1         1         -0.269         -		0.467	0.447	0.497	0.463	0.282	0.14	-0.21	E.0-	0.196	-0.25	0.326	-0.17
Plane     Direction       1     1     2     1     1     1     0.889       -1     1     2     1     1     1     0.889       1     -1     2     1     -1     1     0.889       1     -1     2     1     1     1     0.889       1     -1     2     1     1     1     0.202       1     2     1     1     1     1     0.239       -1     2     1     1     1     1       2     1     1     1     1     0.239       2     1     1     1     1     0.281       2     1     1     1     1     0.269       2     1     1     1     1     0.259       2     1     1     1     1     0.65       2     1     1     1     1     0.65													
Plane     Direction       1     1     2     1     1       -1     1     2     1     1       1     -1     2     1     1       1     1     2     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       2     1     1     1     1       2     1     1     1     1       2     1     1     1     1       2     1     1     1     1		0.889	-0.446	-0.202	0.841	-0.165	0.239	-0.581	0.115	-0.087	-0.259	0.65	0.282
Plane     Direction       1     1     2     1     1       -1     1     2     1     1       1     1     2     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       1     2     1     1     1       2     1     1     1     1       2     1     1     1     1       2     1     1     1     1       2     1     1     1     1													
Plane         Directic           1         2         1         1           -1         1         2         1         1           -1         1         2         1         1           1         -1         2         1         1           1         2         1         1         1           1         2         1         1         1           1         2         1         1         1           2         1         1         1         1           2         1         1         1         1           2         1         1         1         1         1           2         1         1         1         1         1         1           2         1         1         1         1         1         1         1           2         1         1         1         1         1         1         1	u	-1	١	1	1	١	-1	1	١	1	1	-	1
Plane         Dire           -1         1         2           -1         1         2         1           -1         1         2         1           1         2         1         2         1           1         2         1         1         1           1         2         1         1         1           2         1         1         1         1           2         1         1         1         1           2         1         1         1         1           2         1         1         1         1	ctic	-	7	-	-	7	-	-	-	-	-	-	Ţ
Plane -1 1 2 -1 1 2 -1 2 1 -1 2 1 -1 2 1 -1 2 1 -2 1 1 2 1 1 2 1 1 2 1 1 2 1 1	Dire	~	-	7	~	-	-	~	7	7	~	-	-
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Plane		2	2	2	4	-	-	-	7	-	-	-	7
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0.165 0.25 -0.326 -0.196 0.301

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Alpha twinning direction	1 0 -1	Parallel direction in beta	1.882 0.381 -1.47	Shear plane in alpha	-1 0 1	Parallel plane in beta	0.575 2.0762 1.472

Parallel direction in beta 0.641 1.622 -1.6 Shear plane in alpha 0 -1 1 2 Parallel plane in beta 1.817 0.835 1.6

Alpha twinning direction 0 1 -1 1

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	0.525	-0.24	0.008	-0.77	0-	0.075	0.324	-0.25	0.849	0.008	-0.32	-0.53
	0.526	-0.315	0.008	-0.849	7E-04	-0.075	0.248	-0.324	0.774	0.008	-0.24	-0.525
Direction	1 1 -1	-1 1 1	1 -1 1	1 1 1	1 -1 1	-1 1 1	1 1 -1	1 1 1	1 1 -1	1 -1 1	-1 1 1	1 1 1
	1	1	1	1	0	0	0	0	1	1	1	1
Plane	1 0	1 0	-1 0	-1 0	1 1	1 1	-1 1	-1 1	0 1	0 1	0 -1	0-1

-0.353 -0.651 -0.651 -0.068 -0.186 0.237 0.635 -0.42 -0.42 -0.167

0.821 -0.183

1012 Twinning in Alpha - Resolved Shear Stress on Martensite Shear System II (continued)

	-				2		
Alpha twinning direction	0 -1 1	Parallel direction in beta	1.622 0.6405 1.602	Shear plane in alpha	0 1 -1	Parallel plane in beta	0.835 1.8167 -1.6

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Parallel direction in beta -0.11 2.373 -0.1 Shear plane in alpha

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Parallel plane in beta

2.567 0.085 0.1

Alpha twinning direction

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	0.889	-0.167	-0.42	-0.635	0.068	0.016	-0.237	0.186	0.651	-0.353	-0.183	-0.821
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-0.068 0.061 0.021 0.107

0.412 0.354 0.4 -0.458

0.39 0.461

0.421 -0.351

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### Prism Slip in Alpha - Resolved Shear Stress on Omega Phase

0	1	1	0		
Alpha slip direction 1 1-2	Parallel direction in beta	U.ZD -U.ZD -3.U/ Slip plane in alpha	1 -1 0	Parallel plane in beta	1.241 -1.241 0.13

		•		•		r	
Alpha slip direction	1 -2 1 0	Parallel direction in beta	1.732 -1.732 1.732	Slip plane in alpha	-1 0 1 0	Parallel plane in beta	-0.75 0.75 1.472

1	0	eta			0		
nc	-	in b	1.3	a	-	beta	1.6
directio	٢	rection	. 392	in alph	-	ane in	0.49 ·
oha slip	-2	rallel di	.992 1	p plane	0	rallel pl	- 191
Alp		Ра	7	Sli		Ра	0

Vectors of transformation shear systems Plane Direction

Orientation 1 (parallel beta phase plane and direction)

-0.246	-0.5	54	0.2	<b>`</b>	1-1	5	2	7
0.246	0.5	54	-0.2	1	1-1	1	5	-2
0.492	1	08	-0.5	1	1-1	2	-	-

Orientation 2 (narallel heta nhase nlane and direction)

1         1         1         1         1         0.073         0.465           2         -1         -1         1         -0.073         -0.465         -0.465	-1 2	-	~	•	0 147	0 783	0.93
2 -1 -1 1 1 -0.073 -0.39 -0.465	- <del>-</del>	·   -	-	. –	0.073	0.392	0.465
	2 -1	-	٢	1	-0.073	-0.39	-0.465

	-	_	_
	0.372	0.515	0.142
	0.442	0.058	-0.38
וום מווח מוובכיווסוו)			
וומסכ הומ	-0.069	0.456	0.525
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### Basal Slip in Alpha - Resolved Shear Stress on Omega Phase

[	0				-		
Alpha slip direction	1 1 -2	Parallel direction in beta	0.26 -0.26 -3.07	Slip plane in alpha	0 0 0	Parallel plane in beta	1 1 0

Alpha slip direction	-2 1 1 0	Parallel direction in beta	-1.992 1.992 1.3	Slip plane in alpha	0 0 0	Parallel plane in beta	1 1 0
Alpha slip direction	1 -2 1 0	Parallel direction in beta	1.732 -1.732 1.732	Slip plane in alpha	0 0 0	Parallel plane in beta	1 1 0

shear systems	•
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Vectors of	Plane

Orientation 1 (parallel beta phase plane and direction)

	7	` `	- - -	- - -
3	0.413		)	
3	0.413	-1 1 0.413	1 -1 1 0.413	-1 1 -1 1 0.413

Orientation 2 (narallel heta nhase nlane and direction)

וומוופו טפומ טוומצב טומוים מווט טוופכווטון)		1 1 1 -0.58	1 1 1 -0.58 -0.853	
מומוופו טפומ טוומצפ טומוופ מווט טוופטו	1 1 1 0	1 1 1 -0.58	1 1 1 -0.58	
טוופווומווטוו ב (נימ	1 -1 2 -1	2 1 1 -1	1 2 -1 -1	

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ion 3 (parallel beta phase plane and direction)	2 1 1 1 1 -0.331 0.143	1 1 1 1 -0.082 -0.567 -0.567	-1 1 1 1 0.249 0.71	
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Orient	- - -	1 -2	2 -1	

		-	2		5	2			
1	2		7	-	-		0.331	-0.19	-0.143
-1 2	1		-	-	1		0.249	0.481	-0.71
-2	-		7	-	1		-0.082	0.674	-0.567

## 1011 Twinning in Alpha - Resolved Shear Stress on Omega Phase

2	1			1		
Alpha twinning direction 1 0 -1	Parallel direction in beta	3.013 1.512 -1.47	Shear plane in alpha	-1 0 1	Parallel plane in beta	-0.087 1.4134 1.472

ion	1 2	eta	2			_	2
winning direct	0	l direction in b	3.013 1.47	olane in alpha	- 0	I plane in beta	-0.087 -1.4
Alpha t	-	Paralle	1.512	Shear p	-	Paralle	1.413

<u>ا - - - - - - - - - - - - - 1.413</u> Vectors of transformation shear systems Plane Direction

Orientation 1 (parallel beta phase plane and direction)

2 -1 1 -1 1 3E-04
1 1 1 -1 1 0.008 -0.592
1 2 1 -1 1 0.008 0.008 0.008

Vacitoria b с С Oriontotion

	-0.17	0.012	0.182	
	-0.27	-0.58	-0.31	
iase plane and direction)	-0.269	0.313	0.582	
la pr				
De	-	-	1	
ralle	-	-	-	
(pa	ŗ	-	-1	
	2	1	-1	ľ
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	-0.8	0.036	0.279	а а
3 (parallel beta phase plane and directio	1 1 1 1 -0.501	1 1 1 1-0.268	1 1 1 0.232	
Orientation 3	-1 -1 2	1 -2 1	2 -1 -1	

	-0.894	-0.139	0.755
	-0.5	-0.23	0.268
וום מווח מוובטיוטו)			
ומסכ הומ	-0.796	-0.832	-0.036
ש			
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מווסו			1
	-	2	-
5	1	-	-2

# 1011 Twinning in Alpha - Resolved Shear Stress on Omega Phase (continued)

	2				1		
Alpha twinning direction	0 -1 1	Parallel direction in beta	2.753 1.7716 1.602	Shear plane in alpha	0 1 -1	Parallel plane in beta	0.172 1.1538 -1.6

	2				-		
Ipha twinning direction	1 -1 0	arallel direction in beta	3.504 1.021 0.13	thear plane in alpha	-1 1 0	arallel plane in beta	-0.58 1.904 -0.13
A		ш.		0)		ш	

Alpha twinning direction	-1 1 0 2	Parallel direction in beta	1.021 3.504 -0.1	Shear plane in alpha	1 -1 0 1	arallel plane in beta	1.904 -0.58 0.1
Alpha tv	-	Parallel	1.021	Shear p	-	Parallel	1,904

Vectors of transformation shear systems Plane Direction Orientation 1 (parallel beta phase plane and direction)

0.376	-0.218	-0.594
0.376	0.594	0.218
-0.375	0.217	0.592
١	1	-
1 -1	1-1	1 -1
2	-	7
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-1	Ņ	Ţ

Orientation 2 (parallel beta phase plane and direction)

	0.418	-0.157	-0.574	
	0.418	0.574	0.157	
מווכ מוומ מווכרווטוו)	2	2	2	
וומסכםוו	-0.17	-0.182	-0.012	
מל				
ומ	1	1	-	
alla	-	-	-	
- (pai	-	-	7	
7 11	2	1	-	
ומוור	Ļ.	-	N	
	-	2	-	

Orientation 3 (parallel beta phase plane and direction)

	)	2001	5	5	2	5 0 0 0 0		
-1 -1 2		-	-	1		-0.894	-0.4	-0.377
1 -2 1		-	-	1		-0.755	-0.62	0.166
2 -1 -1		-	-	1		0.139	-0.21	0.543

	-0.404	0.215	0.619
	-0.38	-0.54	-0.17
וום מווח מוובריותוו)			
ומסכ הומ	-0.559	-0.279	0.28
קא			
20	1	1	1
	-	-1	7
מש	-	-	7
t			
	2	1	7
וומ	-	2	-
5	-	-	-2

## 1012 Twinning in Alpha - Resolved Shear Stress on Omega Phase

~	•			2		
Alpha twinning direction	Parallel direction in beta	1.882 0.381 -1.47	Shear plane in alpha	-1 0 1	Parallel plane in beta	0.575 2.0762 1.472

Alpha twinning direction	-1 0 1 1	parallel direction in beta	0.381 1.882 1.472	shear plane in alpha	1 0 -1 2	parallel plane in beta	2.076 0.575 -1.47
Alpha twinning di	-1 0	Parallel direction	0.381 1.882	Shear plane in al	1 0	Parallel plane in I	2.076 0.575

Alpha twinning direction01-1Parallel direction in beta0.6411.622Shear plane in alpha0-11Parallel plane in beta1120	0.1 0.0.0 /10.1
--------------------------------------------------------------------------------------------------------------------	-----------------

U.575 2.0762 Vectors of transformation shear systems Plane Direction

Orientation 1 (parallel beta phase plane and direction)

-0.446	-0.165	0.282
0.009	0.005	0-
0.009	0.004	-0.005
1	1	1
1 -1	1-1	1 -1
2	1	-
-1 1	-2 -1	-1 -2

Orientation 2 (parallel beta phase plane and direction)

Paramet Deta priase priare and unection           -1         1         1         -0.321           -1         1         1         -0.255           -1         1         1         0.096	2         -1         1         1         -0.321           2         -1         1         1         -0.325           1         -1         1         -0.225           1         -1         1         0.096	ation 2 (paramet beta priase plane and unection) 1 2 -1 1 1 -0.321 2 -1 -1 1 1 0.096
Paranier Ucta pr -1 1 1 -1 1 1 -1 1 1	2 - 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	ation 2 (paramet beta pr 1 2 -1 1 1 2 -1 -1 1 1 2 -1 -1 1
-1 1 -1 1 -1 1	2 -1 1 -1 1 -1 1 -1 1	ation 2 (parallet t 1 2 -1 1 2 -1 -1 1 2 -1 -1 1
		ation z (parc 1 2 -1 2 -1 -1 2 -1 -1
		2 -1 1 2 - 1 1 2 -

Orientation 3	(par	allel	beta	pha	ase plane and direction)		
-1 -1 2	-	-	-	-	-0.793	-0.75	-0.889
1 -2 1	۲	-	1	-	-0.116	-0.16	-0.65
2 -1 -1	۲	-	1		0.677	0.59	0.239

	-0.841	-0.259	0.581
	-0.79	-0.68	0.116
ווס מוומ מוו סטוול			
ומטר הומ	-0.75	-0.59	0.16
יומ או			
	-1	-1	-1
ושמי	-	-	-
5	2	1	-
וומו	-	2	-
)	-	-	-2

# 1012 Twinning in Alpha - Resolved Shear Stress on Omega Phase (continued)

	1				2		
Alpha twinning direction	0 -1 1	Parallel direction in beta	1.622 0.6405 1.602	Shear plane in alpha	0 1 -1	Parallel plane in beta	0.835 1.8167 -1.6

_	-	٣			2		
direction	0	n in beta	0.13	alpha	0	beta	-0.13
vinning o	7	direction	-0.11	lane in a	ſ	plane in	2.567
Alpha tv	-	Parallel	2.373	Shear p	-	Parallel	0.085

Alpha twinning direction -1 -1 -0 -1 Parallel direction in beta -0.11 2.373 -0.1 Shear plane in alpha	Parallel plane in beta
-------------------------------------------------------------------------------------------------------------------	------------------------

L 0.835 1.8167 Vectors of transformation shear systems Plane Direction

Orientation 1 (parallel beta phase plane and direction)

-1 1 2	<u>.</u>	- -	1	-0.446	0.447	0.447
-2 -1 1		1-1	-	-0.282	0.165	0.282
-1 -2 -1		1 -1	-	0.165	-0.28	-0.165

and direction) orallal bata phace Oriontation 2 (n

Orientation 3 (parallel beta phase plane and direction)

	· ~ ~ /		2			
-1 -1 2	-	-	1	-0.841	-0.46	-0.467
1 -2 1	-	٢	-	-0.581	-0.21	-0.326
2 -1 -1	-	-	1	0.259	0.25	0.14

- 6			_
	-0.463	-0.25	0.213
	-0.47	-0.14	0.326
ומטר טטמו	-0.889	-0.239	0.65
קא			
2	1	1	1
2 I N	-	-	7
2	7	-	-
	2	1	1
	1		'
	۰	-1	?

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