Application of Bayesian Neural Network Modeling to Characterize the Interrelationship between Microstructure and Mechanical Property in α+β-Titanium Alloys

DISSERTATION

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Abstract

Titanium alloys, especially $\alpha+\beta$ titanium alloys are used extensively in the aerospace industry because of their attractive balance of properties. The mechanical properties of these materials are very much sensitive to their microstructure. Microstructure in these alloys can be controlled essentially through alloy composition and various thermomechanical processing routes. Microstructures in these alloys are characterized in terms of size, distribution and volume fraction of both $\alpha$ (HCP crystal structure) and $\beta$ (BCC crystal structure) phases. The above-mentioned features can coexist and span different length scales. The interrelationships between the microstructure and mechanical properties are characterized qualitatively in the literature. Physics based models are difficult to implement due to the presence of a wide variety of microstructural features with different length scales and mutual interaction of these features. The modeling of such properties is much more complex when composition is added as an additional degree of freedom.

In this work neural network models with a Bayesian framework have been employed to characterize the microstructure and mechanical property interrelationships in $\alpha+\beta$ Ti alloys based on Ti-$x$Al-$y$V ($4.76<x<6.55;\ 3.30<y<4.45$) with controlled variations in interstitial oxygen (O) and Fe ($0.07$ wt% $O<0.20;\ 0.11<$wt% Fe$<0.41$). These alloys are subjected to various heat treatments and thermomechanical processing
conditions such as β annealing and α+β processing to obtain a range of microstructure and mechanical properties. The important microstructural features in α+β processed α+β titanium alloys are equiaxed alpha grain size, volume fraction of equiaxed alpha grains, width of the α lamellae in transformed β matrix and important features in β heat treated α+β titanium alloys are size of α colony, width of the α lamellae, prior β grain size, volume fraction of colony and grain boundary α thickness. A database is populated with the above-mentioned quantified microstructural information, composition and mechanical properties. The mechanical properties predicted in this study are tensile properties and fracture toughness.

Based on the controlled virtual experiments conducted using neural networks on α+β processed alloys suggested important microstructural features that will affect tensile properties are size of the equiaxed alpha grain and volume fraction of equiaxed alpha. The controlled virtual experiments on β heat-treated alloys suggested important microstructural features such as width of the α lamellae, α colony size and prior β grain size have negative influence on tensile properties.

The virtual experiments conducted on alloys which are processed in the α+β phase field suggested that the size of the equiaxed alpha is an important variable which increases the fracture toughness. In β-processed alloys, important microstructural features such as size of the α colony decrease the fracture toughness while width of the α lamellae and prior β grain size increase the fracture toughness.
The alloying elements such Al, O and Fe improve the yield strength of both $\alpha+\beta$ processed and $\beta$ processed $\alpha+\beta$ titanium alloys. The O and Al have negative influence on fracture toughness while Fe has positive influence on fracture toughness.

The examination of the region beneath the fracture surface of $\alpha+\beta$ processed alloy suggested occurrence of the microcracks within the equiaxed alpha particle clusters. The frequency of occurrence of the microcracks is increased when two neighboring equiaxed alpha grains have common or near common basal plane. The detailed dislocation analysis on regions near the microcrack indicated presence of extensive basal slip.
Dedicated to my family and friends
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Chapter 1 Introduction

Titanium alloys, especially $\alpha+\beta$ Ti alloys, are used in various applications related to aerospace structures and engines. These alloys are ideal candidates for aerospace applications because they possess high specific strength, high temperature capability, excellent corrosion resistance and good damage tolerance [1-5]. Other non aerospace applications of $\alpha+\beta$ Ti alloys include the energy industry [6]. Both $\alpha+\beta$ Ti alloys and majority of $\beta$ Ti alloys are also popular in the medical field because of their high strength, low modulus, good tissue tolerance and excellent corrosion resistance [7, 8]. In titanium alloys, the mechanical properties are very sensitive to their chemistry and microstructure. So increased uses of these alloys in the above-mentioned applications require an increase in understanding of the microstructure and mechanical property interrelationships. In titanium alloys, qualitative interrelationships between microstructure and properties have been established from the vast resources of experimental data. However, the available data is often confusing and difficult to interpret. It is evident from the literature on titanium alloys that small colonies of $\alpha$ or small $\alpha$ laths increase the yield strength but no quantitative scientific treatment has been conducted to assess its effect. It is also understood from the legacy data that the fracture toughness of a material can be improved by designing the microstructure so that the
propagating crack is deflected, but this is only a qualitative assessment. The extent of the improvement in toughness is hard to explain unless mechanistic based models are available. Mechanical properties of titanium alloys such as tensile strength, elongation, toughness and fatigue life cannot be predicted from dislocation theory.

Any predictive model that correlates microstructure to mechanical property in titanium alloys should take microstructural features into account. However microstructural features in titanium alloys are complex, span multiple length scales and are interdependent. This would make implementation of physics based models to predict interrelationships between microstructure and mechanical properties in titanium alloys difficult. Neural networks, a class of non linear data modeling tools can be used to study microstructure and mechanical property interrelationships in titanium alloys because neural networks can handle large number of input variables with complex interdependencies. Neural network do not take into account of prior knowledge of physics into the models. However ideas about the knowledge of microstructure can be included into quantitative models. Neural networks can be used as short-term alternatives until more robust physics based models are developed.

Attempts have been made to predict the mechanical properties of $\alpha+\beta$ titanium alloys in terms of composition and processing parameters using neural networks [9]. However, the above efforts have ignored the incorporation of microstructural features into neural network models. Other researchers employed microstructural features into neural network models to predict properties however their study is limited to specific alloy system with limited range in mechanical properties [10, 11]. The use of
microstructural data in neural networks is limited by the lack of quantitative microstructural data. Moreover, neural networks require high-fidelity data to predict the interrelationships between microstructure and mechanical property accurately. The quantitative characterization of α+β titanium alloys is difficult because they require use of different length scale characterization tools anywhere from optical microscope to transmission electron microscope.

This work describes the application of a rules-based approach as a solution to property modeling, and includes the population of a database containing information regarding alloy composition, quantified microstructure, tensile and fracture properties for a set of α+β processed and β-annealed α/β Ti-alloys based on Ti-xAl-yV (4.76<x<6.55; 3.30<y<4.45) with controlled variations in interstitial O and Fe (0.07<wt%O<0.20; 0.11<wt%Fe<0.41). The composition of the above alloy systems is a slight extension to composition of commercially available α+β Ti alloy Ti-6Al-4V. The above-mentioned alloys were subjected to α+β processing and β-annealing treatment to manipulate microstructure and get an extended range in mechanical property values. Tensile and fracture toughness properties were predicted for both α+β processed and β-annealed microstructure. In real thermo-mechanical treatments of titanium alloys it is difficult to control certain microstructural features without affecting others. For example, in a β annealed α+β titanium alloy, it is not possible to control the thickness of grain boundary α without altering the width of the α lamellae. This makes the interpretation of the effect of single microstructure on properties difficult because contributions to properties come from various microstructural features. Neural networks can handle this situation through
certain virtual experiments where certain microstructural features are allowed to vary while keeping the remaining microstructural features to an average value. In the current work, virtual experiments are conducted based on the developed neural network models to illustrate the contributions of the composition and various microstructural features to the mechanical properties.

The models developed using neural networks are not physics based so they require metallurgical explanation of the trends observed in the predicted mechanical properties. Interpretation and validation of the trends observed in neural network property modeling of titanium alloys requires critical experiments. This research explores the effect of microstructural features on fracture toughness of $\alpha+\beta$ processed titanium alloys. This study was undertaken primarily to study deformation mechanisms related to titanium alloys.

The following chapter on literature review makes an attempt to shed the light on structure of titanium, thermo-mechanical processing of $\alpha+\beta$ titanium alloys, microstructural evolution in $\alpha+\beta$ titanium alloys, and the legacy understanding of the effect of composition and microstructure on mechanical properties.
Chapter 2 Literature Review

2.1 Introduction

This chapter gives a brief overview on the physical metallurgy of titanium, thermomechnical processing routes related to $\alpha+\beta$ Ti alloys, and a summary of the legacy understanding on interrelationships between microstructure and mechanical properties in titanium alloys. This fundamental understanding of titanium metallurgy is useful in analyzing trends observed in phenomenological models such as neural networks.

2.2 Physical Metallurgy of Titanium

Titanium is an allotropic material with a room temperature $\alpha$ phase with a HCP (Hexagonal Close Packed) crystal structure ($c/a=1.587$; $c=0.468$ nm, $a=0.295$ nm) as well as a high temperature $\beta$ phase that has a BCC (Body Centered Cubic) crystal structure ($a=0.332$ nm). Figure 2.1 shows two different crystal structures of titanium with important crystallographic planes and directions outlined [12]. Pure Ti undergoes a phase transformation at $882^\circ$ C; this transformation temperature is called $\beta$ transus. The $\beta$ transus temperature varies depending on the presence of interstitial and substitutional elements. Alpha stabilizers such as Al and O raise the $\beta$ transus temperature, i.e., stabilize the $\alpha$ phase while beta stabilizers such as V and molybdenum decrease the $\beta$ transus temperature.
2.2.1 Classification of Titanium Alloys

Titanium alloys are classified as $\alpha$, $\alpha+\beta$ or $\beta$, based on their composition. Titanium is typically alloyed with alpha stabilizers such as Al, O, N or C, beta stabilizers, such as Mo, V, Nb, Ta (Isomorphous), Fe, W, Cr, Si, Ni, Co, Mn, and H (eutectoid) or neutral elements such as zirconium. The relative stability of the $\alpha$ phase in titanium alloys is specified in terms of the Al equivalent or the O equivalent [13].

\[
\text{Al Equivalent} = \%\text{Al} + (1/3\times\%\text{Sn}) + (1/6\times\%\text{Zr}) + (10\times\%O)
\]

\[
\text{Oxygen Equivalent} = \%\text{O} + (1.2 - 2.0\times\%\text{N}) + (0.67\times\%\text{C})
\]

The relative stability of the $\beta$ phase is expressed in terms of the molybdenum equivalent [13].

\[
\text{Molybdenum Equivalent} = \%\text{Mo} + (\%\text{Ta}/5) + (\%\text{Nb}/3.6) + (\%\text{W}/2.5) + (\%\text{V}/1.5) + (1.25\times\%\text{Cr}) + (1.25\times\%\text{Ni}) + (1.7\times\%\text{Mn}) + (1.7\times\%\text{Ni}) + (2.5\times\%\text{Co})
\]

Important titanium alloys and their applications are listed in Table 1.

**Alpha and near $\alpha$ Ti alloys:** Alpha alloys consist of commercially pure titanium or titanium with small amounts of alpha stabilizers such as Al, O, and Sn added to it. These alloys do not respond to heat treatment. Small amounts of beta stabilizers are added to make these alloys partially respond to heat treatments and to enhance processing characteristics. Typical microstructural features observed at room temperature are equiaxed alpha, widmanstätten alpha, and martensitic alpha. Presence of the above-mentioned features depends on cooling rate. These alloys are preferred for elevated
temperature applications because of their good mechanical properties combined with superior creep resistance at elevated temperature [14].

**Alpha+beta Ti alloys:** $\alpha+\beta$ alloys contain approximately 4-6% $\beta$ stabilizers such as Mo and V. These alloys possess higher strength than near $\alpha$ alloys and respond to the heat treatment. They have a good balance of properties and a large processing window, which allows for microstructure manipulations. Properties of $\alpha+\beta$ alloys depend on composition, relative proportion of $\alpha$ and $\beta$ phases, prior thermal history, and thermomechanical treatment. Common heat treatments in these alloys include $\beta$ processing which produces colony / basketweave type microstructure and $\alpha+\beta$ processing which create bimodal microstructure. $\alpha+\beta$ processed alloys show high ductility while $\beta$ processed alloys possess good damage tolerance properties such as fracture toughness. Ti-6Al-4V, the most versatile $\alpha+\beta$ alloy has a good balance of strength, ductility, fatigue and fracture toughness [12].

**Beta Ti alloys:** Beta alloys contain enough beta stabilizers, so that the $\beta$ phase is stable at room temperature. According to Williams et al. [12], beta alloys are defined as alloys that retain the $\beta$ phase under rapid cooling with suppression of the martensite phase [12, 14]. Beta alloys are normally alloyed with two types of alloying elements, namely $\beta$ isomorphous (V, Mo, Ta, Nb) and $\beta$ eutectoid (Fe, Cr, Fe, Mn, Cu). Beta titanium alloys offer good tensile properties, good formability, good toughness but poor creep resistance.
2.3 Phase Transformations in Titanium Alloys

Titanium alloys have complicated phase diagram. Based on the composition and cooling rate we can produce stable and metastable phases in these alloys. The amount and type of phases present at room temperature are determined by cooling rate and composition. Important equilibrium and non-equilibrium phases found in Ti alloys are described in the section below.

2.3.1 Equilibrium Phases in Ti Alloys

**Beta phase:** Beta phase is a high temperature phase of titanium with a BCC crystal structure. This phase is stabilized at room temperature by addition of beta stabilizers such as V and Mo, which lower the β transus. The volume fraction of β phase retained at room temperature is proportional to the amount of beta stabilizers added to the titanium.

**Alpha phase:** Alpha phase is a room temperature equilibrium phase with a HCP crystal structure. Important alpha stabilizers, Al and O strengthen the alpha phase and raise the β transus temperature. When a typical α+β Ti alloy is cooled from β phase field to α+β phase field, the plates of α phase nucleate at prior beta grain boundary. Further reduction in temperature leads to the nucleation of alpha plates at the interface of grain boundary alpha or at the beta grain boundary. These alpha plates grow into the beta grain as a colony of the same variant. This colony of alpha will grow into the beta grain until it is intersected by a colony of different variant. These α colonies consist of plates of alpha with a thin film of beta phase between them. The size of these colonies is determined by the size of the beta grains. The morphology of the α phase depends on cooling rate and
the chemical composition. During phase transformation the Burgers orientation relationship \( \{110\}_\beta \parallel (0001)_\alpha \) and \( <111\>_\beta \parallel <11-20>\_\alpha \) between the \( \beta \) and \( \alpha \) phase will be maintained [15, 16]. The schematic of the nucleation and growth of the \( \alpha \) phase in Ti-6Al-4V is shown in Figure 2.4 [12, 14, 17, 18].

**Ti\(_3\)Al \( (\alpha_2) \):** Ti\(_3\)Al is an ordered intermetallic phase commonly found in titanium alloyed with Al. Formation of this phase is promoted when the presence of Al is more than 5 wt%. This phase precipitates when the Ti alloys are aged below the Ti\(_3\)Al solvus temperature. Oxygen aids the formation of this phase. This intermetallic phase promotes planar slip, so it has a positive influence on tensile strength and a negative influence on fracture toughness.

**TiAl \( (\gamma) \):** This is also an ordered phase with L1\(_0\) crystal structure. This phase commonly found in alloys with high Al content.

### 2.3.2 Non equilibrium Phases

**Hexagonal Martensite \( (\alpha') \):** This is a common type of martensite with a hexagonal crystal structure. It normally forms in dilute alloys with high cooling rates from the \( \beta \) phase field. It maintains its Burgers [15] orientation relationship with the \( \beta \) phase.

**Orthorhombic Martensite \( (\alpha'') \):** Orthorhombic martensite forms under rapid cooling when large amounts of beta stabilizers are present.

**Omega phase \( (\omega) \) [19]:** Omega phase forms in titanium alloys that contain high amounts of \( \beta \) stabilizers. This phase has a hexagonal crystal structure. Omega phase can be identified as athermal omega or isothermal omega depending on the heat treatment.
Athermal omega forms under rapid cooling from the beta phase field while isothermal omega forms during the isothermal aging in $\omega + \beta$ phase field.

### 2.4 Processing and Microstructure of $\alpha+\beta$ Ti Alloys

$\alpha+\beta$ titanium alloys are subjected to various heat treatments and mechanical processing routes to achieve fully lamellar microstructure, globular microstructure and bimodal microstructure [12, 20, 21]. This section of the literature review tries to address the microstructural evolution, important processing variables that control the microstructural features and their subsequent effect on mechanical properties.

#### 2.4.1 Beta Annealing (Fully lamellar microstructure)

Complete schematic of this process is described in Figure 2.5 [12, 20, 21]. Important steps in this processing route are (i) homogenization (in $\beta$ - phase field), (ii) deformation (in $\beta$ or $\alpha+\beta$ phase field), (iii) recrystallization (in $\beta$ - phase field) and (iv) annealing. In this processing route, titanium alloys typically homogenized in the $\beta$ phase field which is above the $\beta$ transus temperature and mechanically worked in the $\beta$ or $\alpha+\beta$ phase field. It is common in the industry to mechanically work in the $\beta$ phase field initially because of the low flow stress, and then to deform in the $\alpha+\beta$ phase field to control the beta grain growth [12]. After deformation in step II, titanium alloys are annealed in the $\beta$ phase field and cooled to $\alpha+\beta$ phase field.

Important features of the lamellar microstructure known to affect properties are size of the $\alpha$ colony, thickness of the $\alpha$ lath and thickness of the $\alpha$ at the prior $\beta$ grain boundary. Typical microstructures of the beta annealed Ti-6Al-4V are shown in Figure
2.6 and Figure 2.7 and important features are outlined in these figures. Figure 2.6 represents an optical micrograph, which shows prior beta grain decorated by grain boundary α and colonies of α within that grain. Figure 2.7, a back scattered electron image shows the colonies of alpha in the β grain. In this figure, the bright phase is β-phase. These features are controlled by the cooling rate from the β phase field. (Step III in Figure 3). As the cooling rate is increased the size of the α colony, thickness of the α lath, and the thickness of the α at prior beta grain boundary are decreased. We can also modify colony microstructure to basketweave type microstructure by increasing the cooling rate. Depending on the heat treatment in step III, secondary alpha from the β phase will precipitate in step IV. The secondary alpha is also an important variable in controlling the mechanical properties of the titanium alloys [12]. For Titanium alloys with Al in it, step IV in the processing route is an important step. In this stage, temperature is an important variable that controls the precipitation of the α2 (Ti₃Al) phase. For Ti-6Al-4V, the solvus temperature of the Ti₃Al is around 550º C [12]. Any annealing treatment performed below this temperature will cause the hardening of the α phase due to Ti₃Al precipitation and annealing above the solvus temperature only serve as stress relieving treatment. In summary, by tuning this thermo mechanical processing route, it is possible get multiple length scale features such as optical scale prior beta grains to nano scale features such as Ti₃Al.
2.4.2 \( \alpha + \beta \) Processing (Bimodal microstructure)

A schematic of the \( \alpha + \beta \) processing is shown in Figure 2.8. The important process steps in \( \alpha + \beta \) processing route of titanium alloys consists of (i) homogenization in the \( \beta \) phase field, i.e., above the \( \beta \) transus (ii) mechanical working in the \( \alpha + \beta \) phase field, i.e., below the \( \beta \)-transus (iii) recrystallization in the \( \alpha + \beta \) phase field and (iv) aging in the \( \alpha + \beta \) phase field \[12, 20, 21\]. The microstructure resulting from the \( \alpha + \beta \) process is characterized by equiaxed alpha in a matrix of transformed beta. Typical bimodal microstructure of Ti-6Al-4V resulting from the above processing route is shown in Figure 2.9 and Figure 2.10. Important microstructural features in bimodal microstructure are (i) size of the equiaxed alpha, (ii) volume fraction of equiaxed alpha, (iii) thickness of \( \alpha \) lath in transformed beta. These microstructural features are outlined in Figure 2.8. The important variable in \( \alpha + \beta \) processing route is cooling rate from the \( \beta \) phase field to \( \alpha + \beta \) phase field. It determines the thickness of \( \alpha \) lamellae and \( \alpha \) colony size. Size of the \( \alpha \) lamellae in Step I controls the size of the equiaxed alpha (\( \alpha_p \)) in the Step III. In Step II the \( \alpha \) lamellae are deformed in the \( \alpha + \beta \) phase field. The degree of deformation is controlled in order to introduce enough stored energy to facilitate the recrystallization of the \( \alpha \) and \( \beta \) phases in Step III. In step III the recrystallization temperature and cooling rate determines the volume fraction of the equiaxed alpha (\( \alpha_p \)). The volume fraction of equiaxed alpha and the size of the equiaxed alpha are normally associated with the beta grain size, which is the most important microstructural variable in bimodal microstructure. The beta grain size is the distance between the primary alpha grains.
This distance between the equiaxed α limits the size of the colony alpha. The cooling rate from the annealing temperature controls the width of the α lamellae while beta grain size controls the width of the α colonies. The cooling rates from the annealing temperature also control the size and volume fraction of equiaxed alpha. At slower cooling rates size of the equiaxed alpha and volume fraction of the equiaxed alpha increases.

During recrystallization, the majority of the α stabilizers, such as Al and oxygen will be partitioned into the equiaxed alpha particles while the majority of the β stabilizers such as V and Mo will be partitioned into the β phase. When the alpha stabilizer, Al is present in large quantities it promotes the formation of Ti₃Al precipitates during aging, which will strengthen the α phase [12, 22]. Ti₃Al precipitation is also aided by the presence of oxygen, which is also a potent alpha stabilizer. The presence of Ti₃Al promotes the planar slip increasing strength however its presence is detrimental to ductility.

2.5 Microstructure and Mechanical Properties of α+β Ti Alloys

2.5.1 Microstructure and Mechanical Properties of Fully Lamellar Microstructures

The intent of this section is to give a legacy understanding on the effect of important features of lamellar microstructure on mechanical properties such tensile, fracture toughness and fatigue.

The important microstructural variables that control mechanical properties of fully lamellar microstructure are (i) α colony size, (ii) α lamellae size, (iii) thickness of α
at β grain boundary, and (iv) β grain size [20, 21]. Alpha colony size has been related to the effective slip length. A schematic showing the effect of slip length on mechanical properties of α+β Ti alloys is shown in Figure 2.11 [12, 21]. It is clear from Figure 2.11 that the reduction in slip length causes an increase in yield strength i.e., microstructures with small α colony size or small α lamellae width have better yield strength. Increases in cooling rate decrease the α colony size contributing to the reduction in the slip length. Significant improvement in yield strength is obtained when the microstructure is changed from colony to martensitic, in which slip length is equal to the width of the individual α lamellae. It should be noted that in such martensitic transformations, the solute content of the individual lamellae is also increased, possibly accounting for a portion of the improvement in yield strength.

Ductility of titanium alloys initially increases with the decrease in colony size and also decreases at higher cooling rates. Experimental evidence reported in the literature suggests that there is a change in fracture mode with an increase in cooling rate. Specifically, the fracture mode changes from transcrystalline at low cooling rates to intercrystalline along the prior beta grain boundaries at higher cooling rates. As suggested in the literature, the intercrystalline mechanism of fracture can be changed to the transcrystalline mechanism with a reduction in beta grain size [12]. There are reports showing improvement in tensile elongation due to a reduction of the prior beta grain size in both lamellar and bimodal type microstructure. This improvement in elongation can be attributed to the prior beta grain size limiting the colony size, thus controlling the slip length of the colony [23].
Continuous alpha present at the prior beta grains also affects mechanical properties. It has been suggested that plastic deformation occurs preferentially at the prior beta grain boundaries, causing the cracks to nucleate at the prior beta grain boundary [24]. Solute partitioning causes the difference in strength between the continuous alpha at prior beta grain boundary and the matrix. This difference in strength can be attributed to the decrease in ductility [12]. The effect of grain boundary alpha can also be associated with the slip length. This slip length will vary with the orientation of the beta grains with respect to the direction of testing. Improvements in ductility and HCF (High Cycles Fatigue) strength were observed when the grains were oriented in such a way that they have less slip length [24].

Experimental evidence [12] suggests that the HCF fatigue strength is related to colony size. As the size of the colony is increased the HCF strength is reduced. This can also be explained in terms of the slip length. Increases in cooling rates decreases the colony size thus decreasing the slip length and increasing the HCF strength [21, 25]. In literature, it has been suggested that the nucleation of fatigue cracks in colony microstructures varies with the cooling rate. At slow and medium cooling rates, the fatigue crack nucleates at shear bands or at the intersection of the shear bands with adjacent alpha colony boundary. At high cooling rates, the fatigue crack normally nucleates at the longest and widest α lath [12]. Fatigue cracks also occasionally originate at the continuous alpha at prior beta grain boundaries. However examination of the fracture showed fracture along the beta grain boundaries [26].
It is reported in the literature [12, 26] that the fine lamellar microstructure has better propagation resistance to microcracks, when compared to coarse lamellar microstructure. In coarse lamellar microstructure fatigue cracks nucleated at the slip bands propagate quickly, while in fine lamellar microstructure, cracks nucleated on long alpha plates propagate slowly as their path is deflected when they encounter colony boundaries. It has been suggested that the colony boundaries and continuous alpha boundaries act as obstacles to crack propagation [12]. An increase in the cooling rate results in increased density of the colony boundaries. Fine lamellar microstructure has more colony boundaries, which can act as obstacles to crack propagation.

Fracture toughness dependence on microstructure comes from two contributions: ductility and roughness of the crack front profile. As the size of the colony is increased, the fracture toughness of \(\alpha+\beta\) titanium alloys increases because the contribution from the roughness of the crack front profile dominates the ductility term. Fracture toughness of the \(\beta\)-annealed \(\alpha+\beta\) titanium alloys is increased by a decrease in the cooling rate, which increases the size of the alpha colony [12, 27]. Hall and Hammond showed enhancement in fracture toughness with increase in inter \(\alpha\) platelet distance and \(\alpha\)-lath thickness. Experimental data on fracture toughness of Ti-6Al-4V suggests that the fine lamellar structure is inferior to the coarse lamellar structure with regards to this property. Niinomi et al. [28], emphasized the effect of the prior beta grain size on fracture toughness of fully lamellar microstructure. They concluded that the large prior beta grain size improves the fracture toughness properties because it has more sub-alpha colonies. These sub-alpha
colony boundaries are known to deflect the crack path and increase the tortuosity of the crack contributing to the higher fracture toughness values [28, 29].

2.5.2 Microstructure and Mechanical Properties of Bimodal Microstructure

The important microstructural parameters known to affect mechanical properties in bimodal microstructure are (i) volume fraction of equiaxed alpha (αp), (ii) size of the equiaxed alpha (αp) and (iii) thickness of the α lath in the transformed beta. The size of the equiaxed alpha particle is associated with the beta grain size, which is the distance between the alpha particles. As mentioned in the literature [12], the beta grain size in commercially processed bimodal microstructure is typically around 30-70 µm, which is very small when compared to the beta grain size of β-annealed microstructure. The colony size in the bimodal microstructure is nominally equal to the size of the beta grain, which is also small compared to that of beta-annealed microstructure. This small colony size corresponds to small slip length. Small slip length in bimodal structures contributes to better yield strength, and higher ductility than the beta annealed fully lamellar microstructure for a similar cooling rate.

Another important effect observed in bimodal microstructure is the alloy element partitioning, which scales with the volume fraction of the equiaxed alpha. During alloy element partitioning, alpha stabilizers are partitioned into the equiaxed alpha particles causing a reduction in basic strength of the α lamellae in the transformed beta. The effect of alloy element partitioning on ductility, propagation of microcracks, and macrocracks,
and fracture toughness is limited because fracture properties are determined by the \( \alpha \) colony size [12].

It is reported in the literature [20] that the influence of volume fraction of equiaxed alpha on yield strength comes either from the colony size or from the alloy element partitioning effect. Colony size has a predominant effect on the yield strength when the volume fraction of equiaxed alpha is low; the alloy element partitioning effect dominates when the volume fraction of equiaxed alpha is high. High yield strength is obtained when the volume fraction of equiaxed alpha is around 10-20%. It will be clearly demonstrated here that the effect of alloy element partitioning on yield strength comes from the volume fraction of equiaxed alpha. The alloy element partitioning depends on alloy chemistry and also scales inversely with the temperature.

The effect of the volume fraction of equiaxed alpha on the yield stress is shown in Table 2.2. It is clear from Table 2.2 [12] that the yield strength of the IMI 834 in bimodal microstructure is higher than that of the fully lamellar microstructure. This is due to the reduction in slip length in bimodal microstructures compared to that of fully lamellar microstructures. However, the reduction of yield strength of the bimodal microstructure at high volume fraction of equiaxed alpha is due to the large contribution of the alloy element partitioning effect. It is also clear from Table 2.2 that the alloy element partitioning effect is reduced with an increase in temperature.

HCF strength (resistance to crack nucleation) in bimodal structure depends on the volume fraction of the equiaxed alpha. It has been suggested in the literature that fatigue
cracks normally nucleate in the lamellar part of the bimodal microstructure [30]. Crack nucleation at the lamellar parts of the bimodal structure is intuitive considering that the alloy element partitioning made the alpha lamellae weaker.

Experiments conducted by researchers [12] at low stress amplitudes show the decrease in HCF strength. This is attributed to the dominant effect of alloy element partitioning over the lesser positive effect of reduction in the colony size. The contribution of alloy element partitioning to the HCF strength is also less pronounced at higher temperatures because partitioning effect is not significant at high temperature.

Fracture toughness properties of the bimodal microstructure are inferior compared to that of lamellar microstructure. The features of interest are small in bimodal microstructure. Because of small features, crack front profile of the bimodal structure is smooth than the crack front profile of fully lamellar microstructure. Improvement in fracture toughness of bimodal microstructure was observed with the increase size of the equiaxed alpha.

2.5.3 Mechanical Properties of Fully Equiaxed Microstructure

Fully equiaxed microstructures have a high enough volume fraction of equiaxed alpha that individual alpha grains begin to interconnect. The important microstructural feature is size of the equiaxed alpha, which is related to the slip length. The effect of slip length on properties is similar to the effect of slip length on other microstructures such as lamellar microstructures. Fine equiaxed alpha has good yield strength due to small slip
length of fine alpha. HCF strength also decrease with increasing size of the equiaxed alpha [12].

Crack front profile of the macrocracks in equiaxed microstructure is relatively smooth compared to the crack front profiles of the fully lamellar structures. Experimental studies conducted on samples with equiaxed microstructure showed a reduction in crack propagation rate with an increase in equiaxed alpha size [31-34]. Experimental data [12] on fracture toughness suggests that the improvement in fracture toughness is due to the coarse size of the equiaxed alpha. Larger equiaxed alpha size corresponds to rough crack front profile resulting in higher fracture toughness values. This result suggests that the crack front roughness term dominates the inherent crack initiation resistance term.

2.6 Effect of Composition on Mechanical Properties

Apart from microstructural strengthening, titanium alloys can also be strengthened by solid solution and precipitation strengthening [12, 35]. But it is difficult to understand the contribution of solid solution strengthening to the mechanical properties in α+β Ti alloys quantitatively as it also involves contribution from the microstructural features. Virtual experiments through neural networks can isolate the contributions of composition to the mechanical properties.

When present as an interstitial impurity, oxygen, a potent alpha stabilizer, can increase the strength of titanium alloys. The effect of oxygen on strength in pure Ti is shown in Figure 2.13 [36]. It increases the yield and tensile strength in pure titanium. In
literature, it has been suggested that the oxygen strengthens the alpha phase and impedes cross slip in titanium alloys [37, 38]. Oxygen strengthens the alpha phase because it promotes the formation of the Ti$_3$Al ($\alpha_2$) when sufficient amount Al is present in the alloy. Presence of Ti$_3$Al ($\alpha_2$) causes the planar slip because $\alpha_2$ particles are coherent and are sheared by moving dislocations. Planar slip contributes to the increase in the yield strength and the reduction in fracture toughness in titanium alloys [39]. Fracture toughness of titanium alloys is greatly improved with the reduction in the amount of oxygen. The effect of oxygen on fracture toughness of diffusion bonded Ti-6Al-4V is shown in Figure 2.14. It is clear from the Figure 2.14 that oxygen has negative effect on fracture toughness.

The addition of Al to titanium increases the alloy’s strength due to solid solution strengthening. Depending on the amount of Al added to titanium, various intermetallics are formed or short range ordering in titanium alloys is occurred. Presence of intermetallic compounds and short-range order cause planar slip during plastic deformation. [39]. This planarity of the slip has positive effect on strength while negative effect on fracture toughness. V and other beta stabilizing elements can be added to increase the solid solution strengthening effect.

**2.7 Data Modeling of Mechanical Properties**

Table 2.3 - shows the influence of various microstructural features on mechanical properties of $\alpha+\beta$ titanium alloys. In Table 2.3, a + sign indicates a positive influence of corresponding microstructural feature on the property; a - sign indicates negative
influence on that property; o indicates no influence on properties. It is clear that the relationship between microstructure and mechanical properties described in the literature thus far is qualitative. The lack of progress in predicting mechanical properties of titanium alloys quantitatively as a function of microstructural features can be attributed to the large number of interdependent variables that influence properties. The development of physical models was not easily undertaken due to the complicated ways in which microstructural features affect mechanical properties. Until now, the development of titanium alloys relied solely on experimental data. There is a need to develop models that illustrate the effect of microstructure on mechanical properties; these models would minimize experimental time and reduce costs.

Attempts have been made in literature to predict properties in titanium alloys using nonlinear data modeling tools such as artificial neural networks, but most of the models rely on processing parameters and do not take microstructural features into account [9, 40]. Kar et al. [10], attempted to use neural networks with microstructural features as inputs and the tensile properties as outputs. This and other prior research shows that neural networks could be very useful in predicting the mechanical properties of titanium alloys.

2.7.1 Neural Networks

Neural networks are non-linear data modeling tools used for empirical regression and classification modeling, essentially designed to eliminate the problems associated with linear regression. Neural networks are flexible enough to derive complex relationship between inputs and outputs. Neural network architecture consists of small
computing elements called neurons that are interconnected, and interact in complex ways. The outcome of neural network training is an optimum set of weights and a function through which inputs can be correlated to outputs. The training of the network involves finding the optimum non-linear relationship between inputs and outputs [41].

2.7.2 Applications of Neural Networks in Materials Science

Neural networks are used in various applications including the various materials spanning from metals to polymers.

Neural Network prediction of Time Temperature Transformation diagrams of Ti alloys [40, 42]: The importance of the prediction of TTT and CCT diagrams is amplified by the fact that experiments to determine them are costly and time consuming. Sha et al. [40], did a comprehensive study of neural network modeling in the prediction of transformation kinetics in Ti alloys, based on the effect of composition. The predicted TTT diagrams for the alloy Ti-6Al-4V agree with the experimental TTT diagrams, which illustrate the ability of neural networks to predict TTT diagrams. The effect of individual alloying elements on TTT diagrams is also studied. The knowledge gained from the neural networks can be used to derive TTT diagrams for the Ti alloys, which do not have experimental TTT diagrams.

Neural network prediction of mechanical properties of Ti alloys: Malinov et al. [43], conducted a study which involves the influence of the alloy composition, processing parameters, and test temperature on the mechanical properties of Ti alloys [9]. The composition included common alloying elements such as Al, Zr, V, Cr, Fe, Cr, and O.
The alloys used in this study are subjected to the various thermo-mechanical processing methods in the $\beta$ and $\alpha+\beta$ phase field. The above heat treatment processes are then digitized for neural network analysis. For a given heat treatment, the cooling rate affects the microstructural features, however the inclusion of cooling rate in the model is not specified. Tensile strength, yield strength, reduction of area, elongation, impact energy, hardness, fatigue strength and fracture toughness were predicted using neural networks. The researchers did not include any of the microstructural features commonly observed in Ti alloys such as beta grain size, $\alpha$ colony size, $\alpha$ lath thickness, and for $\alpha+\beta$ processed features thickness of the $\alpha$ lath, size of the equiaxed alpha and volume fraction of equiaxed alpha. The model can be used to predict the properties listed above, but without being able to input microstructural features, the fundamental understanding of the structure and properties is limited. The authors also developed a method to study functional relationships between inputs and properties [9]. It was observed that Al increases the strength and decreases the impact energy of the alloys, which is consistent with the literature available on Ti alloys.

**Neural network prediction of fatigue stress life of Ti-6Al-4V**: Mcshane et al. [44], showed that the fatigue stress life of Ti-6Al-4V can be predicted using neural networks. They simulated the fatigue stress life diagrams for various inputs such as microstructure, texture, surface treatment and stress amplitude. The introduction of microstructure into the neural networks is digitized and no effort was made to include quantitative features of the microstructure. Although the predicted fatigue stress life diagrams are in agreement with experimental fatigue life diagrams, the accuracy of the model would have increased
with the introduction of quantitative microstructural features as inputs. The developed models explored the possibility of using neural networks to predict the fatigue stress life of materials for which fatigue stress life data is not available in the literature. Fotovati et al. [45], made an attempt to predict fatigue crack growth rates in Ti-6Al-4V. Artymiak et al. [46], also tried using neural networks to predict S-N curves.

**Neural network prediction of isothermal forging of Ti-6Al-4V**: The microstructure developed in isothermal forging of Ti-6Al-4V has been modeled using neural networks [47]. Strain rate, true strain, and temperature of the deformation are used as inputs. Volume fraction of alpha and grain size of the alpha is the predicted outputs. The predicted values for the outputs are within 7% of the experimental values. It can be seen from the analysis that the processing parameters can be optimized to produce the desired microstructure, which in turn can control the mechanical properties.

Neural networks have also been used to model the fatigue crack growth rate in nickel base super alloys [48], creep rupture life of Ni base super alloys [49], fatigue thresholds in Ni-base super alloys [50], phase transformation in steels [51], hardness of Fe [52], toughness of steels, austenite formation of steels [53], and recrystallization in Al-Mg alloys [54].
<table>
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<tr>
<th>$\alpha$ - Alloys</th>
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<tr>
<td>Ti-3Al-2.5V</td>
<td>Hydraulic tubing in Aerospace Industry</td>
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<tr>
<td>Ti-5-2.5</td>
<td>Cryogenic applications</td>
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<td>$\alpha+\beta$ - Alloys</td>
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<tr>
<td>Ti-6Al-4V</td>
<td>Gas Turbine Engines, Airframes</td>
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<tr>
<td>Ti-6Al-6V-2Sn</td>
<td>Landing gear support structures</td>
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<tr>
<td>IMI 550 (Ti-4Al-2Sn-4Mo-0.1Si)</td>
<td>Components for aero-engine and airframes</td>
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<td>Gas turbine engine components</td>
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<tr>
<td>Ti-6Al-2Sn-4Zr-2Mo-0.1Si</td>
<td>Baldes, discs and rotos, and high pressure compressors</td>
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<tr>
<td>$\beta$ - Alloys</td>
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<td>Springs, torsion bars, fasteners</td>
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</tr>
<tr>
<td>Ti-6Al-2Sn-4Zr-6Mo</td>
<td>Compressor disks, fan blades</td>
</tr>
</tbody>
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Table 2.1 Applications of various Ti alloys
<table>
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<th>Microstructure</th>
<th>Test Temp.</th>
<th>$\sigma_{0.2}$ (MPa)</th>
<th>UTS (MPa)</th>
<th>$\sigma_F$ (MPa)</th>
<th>EI (%)</th>
<th>RA (%)</th>
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<tr>
<td>Lamellar</td>
<td>RT</td>
<td>925</td>
<td>1015</td>
<td>1145</td>
<td>5</td>
<td>12</td>
</tr>
<tr>
<td>Bi-modal (20 vol % $\alpha_p$)</td>
<td>RT</td>
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<td>1100</td>
<td>1350</td>
<td>13</td>
<td>20</td>
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<tr>
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<tr>
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<td>640</td>
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<td>26</td>
</tr>
<tr>
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<tr>
<td>Bi-modal (40 vol % $\alpha_p$)</td>
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Table 2.2 Comparison of tensile properties in bimodal and lamellar structure of Ti-6Al-4V

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<th>$\sigma_F$</th>
<th>HCF</th>
<th>$K_{IC}$</th>
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<td>Small $\alpha$ colonies, $\alpha$ lamellae a</td>
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<td>+</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>Bi-modal structure b</td>
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<td>+</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Small $\alpha$ grain size c</td>
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<td>+</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>Aging ($\alpha_2$), Oxygen</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>Secondary $\alpha$ in $\beta$</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>0</td>
</tr>
</tbody>
</table>

a Compared to coarse lamellar structure  
b Compared to fully lamellar structure with same cooling rate  
c Compared to large a grain size of fully equiaxed structure

Table 2.3 A summary of relationships between microstructure and mechanical properties in $\alpha+\beta$ Ti alloys
Figure 2.1 Unit cell of the Ti (a) HCP and, (b) BCC [12]

Figure 2.2 Schematic of crystal planes and direction of <a> type slip HCP crystal
Figure 2.3 Schematic of the \(<c+a>\) type slip in HCP crystal

Figure 2.4 Schematic of the alpha phase nucleation on prior \(\beta\) grain boundary in Ti-6Al-4V [18]
Figure 2.5 Schematic of the processing route for fully lamellar microstructure [20]

Figure 2.6 Optical micrograph of the β-annealed microstructure of Ti-6Al-4V
Figure 2.7 Back scattered electron image of the β-annealed Ti-6Al-4V

Figure 2.8 Schematic of the α+β processing [20]
Figure 2.9 Optical micrograph of the α+β processed Ti-6Al-4V

Figure 2.10 Back scattered electron image of the α+β processed Ti-6Al-4V
Figure 2.11 Effect of slip length on mechanical properties of α+β Ti alloys [12]
Figure 2.12 Effect of α+β processing parameters on mechanical properties [12]

Figure 2.13 Effect of O concentration on tensile properties of pure Ti [36]
Figure 2.14 Effect of O concentration on fracture toughness of diffusion bonded Ti-6Al-4V [55]
Chapter 3 Experimental Procedures

Introduction of microstructural features related to \( \alpha+\beta \) Ti alloys into neural networks requires a large amount of quantified microstructural and corresponding mechanical property data. However most of the data available in literature on \( \alpha+\beta \) Ti alloys is on composition and processing parameters relating to mechanical properties. There was limited information on the quantified microstructural data relating to mechanical properties. Moreover, microstructural features in \( \alpha+\beta \) Ti alloys are very complex and require special stereological procedures to quantify them. The ability to use microstructural features as inputs in neural networks requires rapid, efficient, and accurate quantitative characterization of microstructural features that are known to affect mechanical properties.

This chapter addresses the experimental methods related to heat treatments and thermo-mechanical processing routes that lead to a variety of microstructures, resulting a wide range in mechanical properties. It also deals with the development of a mechanical property and microstructural database. This database is subsequently used to make predictions of mechanical properties using neural networks. This chapter also deals with characterization tools to study deformation mechanisms in \( \alpha+\beta \) processed \( \alpha+\beta \) alloys. To summarize, this chapter talks about (i) Microstructural evolution in \( \alpha+\beta \) Ti alloys, (ii)
Database development, which is comprised of composition, microstructural data and mechanical properties, (iii) Implementation of neural networks to predict tensile and fracture toughness, (iv) Electron back scattered diffraction (EBSD) study of α+β processed fracture toughness samples and (v) Study of deformation mechanisms in α+β processed fracture toughness samples using a Transmission Electron Microscope (TEM).

3.1 Microstructural Evolution

The material system of importance in this study is an α+β Ti alloy, Ti-xAl-yV (4.76<x<6.55; 3.30<y<4.45) with controlled variation of interstitial alloying elements O and Fe ((0.07 wt% O< .20; .11<wt% Fe<0.41). There are nine samples in the database with composition in the above-mentioned range; for each of the nine alloy compositions, six samples were exposed to different thermo mechanical processing histories, producing a total of 54 samples. The treatments included α+β processing and β-annealing with different cooling rates. Microstructural evolution in this material system is studied systematically as a function of composition, various heat treatments and thermo mechanical processing routes.

3.1.1 α+β Processing of α+β Ti alloys

A schematic of the typical α+β processing is shown in Figure 3.1[12]. The typical microstructure produced from this processing route is a bimodal microstructure. Chemical analysis of these alloys and the relevant heat treatments are given in Table 3.1. These alloys are homogenized in the β-phase field and forged in the α+β phase field as shown in Step I and Step II, respectively, in Figure 3.1. After forging, these samples are
recrystallized in the $\alpha+\beta$ phase field. The recrystallization temperature for each alloy is calculated based on its composition, using PANDAT software. PANDAT is a software designed based on CALPHAD (CALculation of PHAse Diagram) that calculates the phase diagram of multi-component systems [56]. The recrystallization temperatures of these alloys, along with their corresponding $\beta$-transus temperatures, are given in Table 3.1. The samples are cooled from the recrystallization temperature at targeted cooling rates of 50°F/min, 150°F/min and 400°F/min. However, the recorded cooling rates are actually ~50°F/min, ~100°F/min and ~250°F/min. The actual cooling rates of the samples are given in Table 3.1. Cooling rate from the recrystallization temperature is an important variable in this processing route because it determines the size and volume fraction of the equiaxed alpha. Microstructural evolution in these alloys for different cooling rates is shown in Figure 3.2. As can be seen in Figure 3.2 the slow cooling has resulted in high volume fraction of equiaxed alpha. The final step in the $\alpha+\beta$ processing route is aging, which was done at 1300°F for 2 hrs. A temperature above the Ti$_3$Al solvus was chosen in order to prevent any $\alpha_2$ (Ti$_3$Al) precipitates from forming. Ti$_3$Al is an ordered phase and its presence in the microstructure leads to planer slip, causing a reduction in ductility. Since the final treatment is conducted above the Ti$_3$Al solvus, it only serves as stress relieving treatment. Microstructure evolution in these alloys as a function of composition is shown in Figure 3.3. It is clear from this figure that composition has affects microstructural evolution.
3.1.2 β Annealing α+β Ti alloys

The schematic of this annealing process is shown in Figure 3.4. Titanium alloys are homogenized and mechanically worked in the β-phase field. The composition and corresponding heat treatment schedule is shown in Table 3.2. The alloys are cooled from their recrystallization temperature, which is in the β-phase field, through the α+β phase field using three different cooling rates. The achieved cooling rates are ~50°F/Sec, 100 °F/Sec and ~250°F/Sec. Variations in cooling rate resulted in the variations in microstructural features. The microstructural evolution in this heat treatment is shown in Figure 3.5. In particular there is a reduction in α colony size, width of the α lamellae and grain boundary α thickness with an increase in cooling rate. After recrystallization, Step III in Figure 3.4, samples were aged at 1300°F for 2 hours. This treatment also included steps to prevent formation of Ti₃Al.

3.2 Database Development

3.2.1 Alloy Composition

A total of nine different Ti alloys (based on Ti-6Al-4V) were produced with deliberate variations in the relative amounts of the individual alloying elements, including interstitial O and Fe. The ranges of the alloy variations in Table 3.3 and Table 3.4 were measured by TIMET using ICP, with reference to two of the standards for the composition of the alloy Ti-6Al-4V. The detailed chemical analysis of the nine samples in the database is shown in Table 3.5. The Al content in the current alloy system was allowed to range well below that of standard Ti-6Al-4V, while the V was allowed a
slightly extended range. The maximum Al content was intentionally kept below the maximum allowed by the standards (see Table 3.3 and Table 3.5) in order to avoid the onset of short range ordering of the HCP α phase which can result in embrittling α2 domains. The O level had a slightly extended range, with one extremely O lean alloy (0.07 wt% - 700 ppm). The Fe level had a significantly extended range with in the nine samples.

Figure 3.6 (a-d) show three-dimensional scatter plots illustrating the distribution of the 54 samples across the compositional space. As can be seen, there is still a large fraction of composition space that remains unrepresented as discrete points in the dataset. We can use a Bayesian neural network to make predictions of the mechanical properties for the missing compositional space or data lean regions. The consequences of such data-lean regions will be discussed.

3.2.2 Mechanical Property Database

Samples of both α+β processed and β-annealed alloys were subjected to room temperature tensile testing and fracture toughness testing. The layout of these samples that were subjected to mechanical testing is shown in Figure 3.7. The samples were chosen so that they were symmetric about the radius of the billet. It is important to note that the samples were sectioned from the billet so that the loading axes of the tensile specimens are parallel to the loading direction of the fracture toughness specimens.
**Tensile Database**

The tensile database consists of yield strength, ultimate tensile strength, % reduction area and % elongation. This database is developed for both $\alpha+\beta$ processed and $\beta$-annealed $\alpha+\beta$ Ti alloys. The distribution of tensile properties in $\alpha+\beta$ processed and $\beta$-annealed Ti alloys are given in Table 3.6. Variation in alloy composition and thermomechanical processing routes resulted in a spread in yield strength of about $\sim271$ MPa in $\alpha+\beta$ processed samples and $\sim265$ MPa in $\beta$ annealed samples. From Table 3.6, it is clear that the yield strength of $\alpha+\beta$-processed samples (bimodal microstructure) is a little higher than the yield strength of $\beta$ processed (lamellar microstructure) material. $\alpha+\beta$ processed samples also have better ductility than $\beta$ annealed samples.

**Fracture toughness**

Fracture toughness of a material $K_{IC}$ characterizes the materials resistance to the fracture. The methodology for plane strain fracture toughness testing of metallic materials is described in ATSM E-399-09 [57]. The specimen geometry used in this work is of the compact tension type. The locations of the compact tension samples in the billet are shown in Figure 3.7. In a typical fracture toughness test, a notch is machined into a compact tension sample and fatigue pre-cracked to create the sharpest possible crack at notch root. To obtain a sharp crack at the notch root, the specimen is typically subjected to a low cycle- high strain rate mode (typically 1000 cycles with a strain of .003). The initial crack length includes the depth of the notch and the length of the fatigue pre-crack [58].

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It is normally not known in advance whether a test is valid for plane strain fracture toughness, \( K_{Ic} \) or mixed mode condition, \( K_Q \). This determination depends entirely on the yield strength and thickness of the material tested. The thickness of the samples tested is decided mainly based on the yield strength of the material. If the materials’ yield strength is doubled, the thickness of the sample required for plane strain conditions is reduced by a factor of four. In this work the thickness of the CT specimen was kept constant for all samples. However, all of the samples did not qualify for the valid plane strain testing conditions. The samples that failed in plane strain conditions in \( \alpha+\beta \) processed samples, had higher yield strengths and were higher in O concentration. The remaining samples failed in plane stress or mixed modes, and possessed low yield strength and low in O concentrations. Thus, the difference in yield strength caused some samples to fail in plane strain mode while the rest failed in the mixed mode. The plane strain fracture toughness is dependent on only metallurgical factors while plane stress fracture toughness depends on metallurgical factors as well as sample geometry. The distribution of the fracture toughness data in \( \alpha+\beta \) processed and \( \beta \) annealed material is given in Table 3.7. The majority of \( \beta \) annealed samples failed in the plane stress condition while one sample failed in plane strain condition.

3.2.3 Development of Microstructural Database Development

Following the tensile tests for both \( \alpha+\beta \) processed and \( \beta \) annealed samples, an undeformed section of the grip from each sample was sectioned using Wire EDM (Electrical Discharge Machining) for metallographic preparation. Samples were also sectioned from the un-deformed sections of the fracture toughness samples of both \( \alpha+\beta \)
processed and β annealed samples. Following traditional metallographic techniques, the samples were characterized using a FEI/Philips Sirion scanning electron microscope (SEM) operating in backscattered mode at 15 kV with a resolution of approximately 3.0 nm. Each image had a resolution of 3872 x 2904 pixels and a depth of 8 bits. The resolution afforded using SEM, especially when compared with other available techniques (e.g., optical micrographs), is of paramount importance to provide the highest fidelity quantified microstructural data to the neural networks. The specimens were imaged at four random locations avoiding overlap and edge effects. Microstructural features present in the four micrographs were quantified using standard stereological techniques developed at The Center for Accelerated Maturation of Materials [59].

Important microstructural features that affect mechanical properties in α+β processed α+β Ti alloys are listed below.

(i) Equiaxed alpha size (equiaxed-α size, µm)
(ii) Volume fraction of equiaxed alpha ($F_{V_{equiaxed-α}}$)
(iii) Volume fraction of total alpha ($F_{V_{total-α}}$)
(iv) Width of the α laths in the transformed β. This is measured based on Gundersen’s derivation ($α$-lath width, µm) [22,23].

The distribution of the quantified microstructural data for α+β processed tensile samples is given in Table 3.8. The scatter plots of the important microstructural features of α+β processed on yield strength is shown Figure 3.8.

Important microstructural features in β-annealed samples were identified from the literature review are listed below:
(i) Width of the $\alpha$ lath

(ii) Colony Scale Factor (CSF), which measure the size of the $\alpha$-colony

(iii) Prior beta grain factor (PBGF), which measure the size of the prior beta grain

(iv) Width of the grain boundary $\alpha$

(v) % Colony

(vi) Volume fraction of total $\alpha$

3.3 **Implementation of Neural Networks**

The experimentally determined data regarding alloy composition, microstructure, and tensile properties were included in a database for additional analysis using Bayesian neural networks. The database was divided into a training set and a test set. The training set is used to train the network while the test set is used to measure the performance of the network. The test dataset represents a fraction of the 54 samples that were not used for the training; in this case, 38 samples were used to train the model and 16 samples were used to test the model. The test set is chosen so that it is partially random and within the limits of the training data. The data is normalized within -0.5 to +0.5. The formula for normalization is given for data variable $x$.

$$x_N = \frac{x - x_{\text{min}}}{x_{\text{max}} - x_{\text{min}}} - 0.5$$

Here, $x_{\text{min}}$ and $x_{\text{max}}$ are minimum and maximum values of the variables in database while $x_N$ is the normalized value of the variable $x$. The database was trained using a neural network with Bayesian architecture, developed by Mackay [13-16]. This probabilistic neural network was used to develop a model, which predicts the
interrelationships between microstructure and properties of the alloy Ti-6Al-4V. The best model is the one with smallest test error. Once developed, the neural network can be used in one of two ways. The first is in the development of a predictive tool, where properties such as tensile and fracture toughness of a test dataset were predicted. The second is to perform virtual experiments. These are usually controlled experiments because the values of given microstructural features can be set to, or held at, given values while mechanical properties are predicted based on those values. In this way, it is possible to obtain information that is not readily available from actual experiments where such control of individual microstructural features is not possible.

3.4 Experimental setup to study the fracture behavior in \( \alpha+\beta \) processed \( \alpha+\beta \) Ti alloys

The fracture behavior of \( \alpha+\beta \) processed \( \alpha+\beta \) Ti alloys is studied using the Scanning Electron Microscope (SEM) and Electron Back Scattered Diffraction (EBSD). The fractured samples are sectioned along the longitudinal portion of the fracture surface. This cross-section of the fractures surface is polished using standard metallographic techniques. After polishing, the cross-section of the fracture surface is imaged with a FEI Siron SEM microscope. This microscope is equipped with a field emission gun and has the ability take high-resolution images (XHD mode). The cross-section of the fracture surface is shown in Figure 3.9. The imaging of the longitudinal cross-section of the fracture surface allows examination of the preference of crack path for various microstructural features as well as of the presence of sub surface microcracks and microvoids. Stereological methods were also developed to measure the area fraction of
certain microstructural features occupied by the crack path. The length of the fracture surface was measured to determine the roughness of the crack front profile. The detailed standard stereological procedures developed to measure the fracture surface are given in Appendix A.

A Philips/FEI ESEM XL -30 FEG scanning electron microscope equipped with the EBSD detector was used to obtain an Electron Back Scattered Diffraction (EBSD) pattern cross-section of the fracture surface. The schematic of the EBSD setup is shown in Figure 3.9 [60]. In this technique, a stationary electron beam hits the sample and electrons diffracted out of the sample create a kikuchi pattern on the phosphor screen. This pattern is analyzed to get the crystallographic information of the sample. We can use this information to measure the orientation of the grains, misorientation across the different grain boundaries and presence of different phases. TSL™ image collection software is used to collect the EBSD data. The collected data is analyzed using the TSL orientation data analysis software. An inverse pole figure map of the cross-section of the fractured sample is shown in Figure 3.10. The inverse pole figure map is color-coded based on the orientation of the grains.

The nature of the subsurface microcracks was studied using the EBSD. These subsurface microcracks typically appear in a cluster of equiaxed alpha particles. The majority of the equiaxed alpha particles on either side of the subsurface microcracks have a significant misorientation with a common basal plane. This is shown in Figure 3.11.
The dual Beam Focused Ion Beam (DB-FIB) is used to section through the microcrack. The schematic of the DB-FIB is shown in Figure 3.12. The DB-FIB has two columns: an electron column and an ion column. Both of these columns make 52° with each other. The ion beam is used to mill the samples while the electron beam is used to image the sample. A small volume of material that contains a subsurface microcrack is extracted using FIB and then is welded to a molybdenum grid. The material is sliced through its volume using a Slice and View technique available in the FIB. This work is conducted primarily to see the microcrack in the three dimensionally. This experiment confirmed the presence of microvoid coalescence.

3.5 Experimentation to study the dislocations in fractured α+β processed Ti Alloys

The deformation mechanisms in α+β processed α+β Ti alloys are studied with the help of the CM 200 Transmission Electron Microscope (TEM). The cross sectional foils for dislocation analysis are prepared at microcracks using FEI’s Helios DualBeam™ focused ion beam (DB-FIB). DB-FIB gives the flexibility for site-specific TEM sample preparation and allows better control of foil thickness. A schematic of the site-specific TEM sample preparation with in situ-lift out is explained in Figure 3.12. In this process the location of TEM foils is identified based on SEM and EBSD analysis. For example, in fracture toughness samples, cross sectional TEM foils are prepared at subsurface microcracks as shown in Figure 3.13 (a). The TEM foil is welded to an Omniprobe™ needle and then transferred it to a molybdenum Omniprobe grid. The foil, which was welded, molybdenum grid is thinned to make it electron transparent. Low kV milling is also employed in order to minimize FIB induced damage. A TEM foil containing the
microcrack is shown in Figure 3.14. The various slip systems and types of dislocations that are present at the sub surface microcracks have been analyzed using the diffraction techniques and two-beam bright field imaging techniques. Two-beam bright field imaging technique is very useful in bringing out the contrast of the dislocations.
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<th>V (wt %)</th>
<th>Fe (wt %)</th>
<th>O (wt %)</th>
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<th>Cooling Rate (°F/min)</th>
<th>Act. CR (°F/min)</th>
<th>Age Temp (°F)</th>
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Table 3.1 Composition and heat treatment of α+β processed samples in database
### Table 3.2 Composition and heat treatment details of β-annealed samples in database

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<th>V</th>
<th>O</th>
<th>Soj Temp (°F)</th>
<th>Cooling Rate (°F/min)</th>
<th>Act. CR (°F/min)</th>
<th>Age Temp (°F)</th>
<th>Age Time (hrs)</th>
<th>β-transus, (°F)</th>
</tr>
</thead>
<tbody>
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### Table 3.3 Composition specification of Ti-6Al-4V alloy

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<th>AMS ELI Plate Specification 4095</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>O</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
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<td>3.5-4.5</td>
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<tr>
<td>MIL HDBK5</td>
<td>965-1138</td>
<td>895-1069</td>
<td>(*) = Gage dependent</td>
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(UTS = Tensile Ultimate Strength, YS = Yield Stress)
Table 3.4 Chemical analyses of standard Ti-6Al-4V and alloys in current database

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<th>Fe (max)</th>
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<td>0.07-0.20</td>
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<table>
<thead>
<tr>
<th>Aluminum wt%</th>
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<th>Iron wt%</th>
<th>Oxygen wt%</th>
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<td>3.380</td>
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Table 3.5 Chemical analysis of nine alloys in the database
Table 3.6 Range in tensile property data in α+β processed and β annealed database

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<th>YS, MPa</th>
<th>UTS, MPa</th>
<th>%RA</th>
<th>% Elongation</th>
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</thead>
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<td>Max</td>
<td>Min</td>
<td>Max</td>
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<td>930</td>
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Table 3.7 Range in fracture toughness data in α+β processed and β annealed database

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<tr>
<td>Input/Output</td>
<td>Min</td>
</tr>
<tr>
<td>--------------------------------------------------</td>
<td>------</td>
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<tr>
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<td>Size of equiaxed alpha, ( \mu \text{m} )</td>
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<tr>
<td>Volume fraction of equiaxed alpha</td>
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<tr>
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<td>UTS, MPa</td>
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Table 3.8 Ranges in microstructural data in \( \alpha+\beta \) processed tensile data
Figure 3.1 Processing route for obtaining bimodal microstructures [12]

Figure 3.2 Effect of cooling rate on microstructural evolution in $\alpha+\beta$ processing (a) slow cooling and (b) fast cooling
Figure 3.3 Microstructural evolution in $\alpha+\beta$ processing as a function of composition (a) high $\alpha$ and High $\beta$ stabilizers, (b) high $\beta$ and low $\alpha$ stabilizers, (c) high $\alpha$ and low $\beta$ stabilizers and (d) low $\alpha$ and low $\beta$ stabilizers

Figure 3.4 Schematic of the heat treatment for producing lamellar microstructure [12]
Figure 3.5 Effect of cooling rate on lamellar microstructures (a) slow cooling and (b) fast cooling
Figure 3.6 3D scatter plots of the composition on yield strength

Figure 3.7 Layout of the fracture toughness and tensile samples in a billet
Figure 3.8 Scatter plots of microstructural data

Figure 3.9 Cross-section of the fracture surface
Figure 3.10 Schematic of the EBSD set up

Figure 3.11 IPF map of (a) when loading direction is sample RD direction and (b) loading direction is sample normal direction
Figure 3.12 Schematic of the DB-FIB TEM sample preparation
Figure 3.13 Preparation of the site specific TEM sample preparation (a) identifying the sites of microcracks, (b) trenching the area (c) welding to an Ominprobe needle and (d) welding to the Omniprobe grid
Figure 3.14 Two beam BF image showing microcrack and $<c+a>$ dislocations
Chapter 4 Neural Networks

4.1 Abstract

Neural networks with a Bayesian framework are used to predict the interrelationship between the microstructure and mechanical properties in α+β Ti alloys. The Bayesian framework is preferred in this work over conventional neural networks because the Bayesian framework quantifies uncertainty in the model prediction with error bars. This chapter talks about the need for neural networks, application and implementation of the Bayesian framework to neural networks, Bayesian inference to control model complexity and various aspects of Bayesian inference in neural network.

4.2 Introduction

Neural networks are non-linear data modeling tools used for empirical regression and classification modeling, essentially designed to eliminate the problems associated with linear regression. Neural networks are flexible enough to derive the complex relationship between the inputs and outputs and powerful enough to capture the interaction among the various input variables, which are interdependent. These models are useful when physics-based models are difficult to implement. Even though these phenomenological models have been used extensively in various applications related to materials science, these models have not traditionally included microstructural features as input variables.
Inclusion of microstructural features, especially in $\alpha+\beta$ Ti alloys, is really important because mechanical properties in these alloys are very sensitive to their microstructure. There is a need to develop models to understand the effect of microstructure on mechanical properties while minimizing the experimental time and cost. These models not only used for short-term predictive capability until more robust physics-based models are developed, but also inform physics-based models with fundamental mechanisms.

There are numerous neural networks tools available in the literature, however in this work, artificial neural networks with Bayesian framework are considered because of their underlying advantages when compared with other neural network models.

4.3 Neural Networks

4.3.1 Schematic of J-I-1 Neural Network Model

Neural network architecture consists of small computing elements called neurons that interconnect and interact in complex ways to define any arbitrary relationship between inputs and outputs. Figure 4.1 shows the schematic of J-I-1 layer neural networks. This is a simple three-layer feed-forward network architecture. This architecture has one input layer, one hidden layer and one output layer. Inputs are connected to the hidden layer through connections called weight functions and information about the inputs is stored in the hidden layer. The hidden node is associated with an activation function such as hyperbolic tangent function. Increasing the number of hidden nodes in a hidden layer increase the complexity of the model. The product of the inputs ($x_j$) and weights ($w_{ij}$) forms an argument to the hyperbolic tangent transfer function (Equation 4.2) [41]. The
hidden layers are connected to the output layer through a linear function (Equation 4.1). Equation and associated weights are studied to reveal the interaction and relationship between inputs and outputs. Varying the weights alters the strength of the hyperbolic function. The shape of the hyperbolic tangent function can be changed with the position in the input space. Therefore it has the ability to capture the relationship between inputs and outputs across the entire span of input space. This is especially important in characterizing Ti alloys because composition and microstructural features are often related to mechanical properties in complex ways. Since the hyperbolic tangent function is non-linear, it can capture interaction between the input parameters. The nature of the interaction can be understood in terms of weights.

\[ y = \sum_i w_i^{(2)} h_i + \theta^{(2)} \]  
\[ h_i = \tanh(\sum_j w_{ij}^{(1)} x_j + \theta_i^{(1)}) \]  

The network mentioned in Equations 4.1 and 4.2 is trained using a training set \(D=\{x^{(m)}, t^{(m)}\}\) through minimization of an objective function \(M(w)\) by adjusting the weights and biases iteratively until the best description of the output is obtained. The objective function with regularization constants \(\alpha\) and \(\beta\) is defined below.

\[ M(w) = \beta E_D + \alpha E_W \]  
\[ E_D(w) = \frac{1}{2} \sum_m \sum_i (t_i^{(m)} - y_i(x^{(m)}; w))^2 \]  
\[ E_W = \frac{1}{2} \sum_i w_i^2 \]
Here, $E_D$ is an objective function that measures how close output $y(x,w)$ is to target $t$, for each input/target pair $\{x,t\}$. $E_D$ increases if the important inputs are omitted from the model. $E_W$ is added to the objective function because it prefers small values of $w$, which makes the neural network model find simpler solutions with less propensity to over-fit to noise in the training data.

### 4.2.2 Neural Networks with Bayesian Framework

Implementation of neural network with Bayesian framework in this research is based on the work of Mackay [61, 62]. His approach to model-fitting is unique because rather than calculating a unique set of weights that minimizes an objective function, he calculates a probability distribution of weights. As a result, his method allows many functions to be fitted or extrapolated into uncertain regions of the input space, whether due to noisy data or lack of data, without affecting the fit in adjacent regions where data are rich and accurate. The error bars are become large when the data is sparse or locally noisy, thus representing both noise in the experimental data and the uncertainty in the prediction.

### 4.2.3 Probabilistic Approach to Neural Network Learning

MacKay [51] gives a probabilistic interpretation to the neural network learning process. He interprets the error function as a probability distribution of noise in his model.

$$P(D | w, \beta, H) = \frac{1}{Z_D(\beta)} \exp(-\beta E_D) \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (4.6)$$

66
This function represents the likelihood in Bayes’ theorem. He interprets sum squared error \( E_D \) in equation (4.4) as Gaussian noise on the target variables where parameter \( \beta \) defines a noise level \( \sigma_n^2 = 1/\beta \). He defines regularizer in terms of log prior probability distribution over the parameters.

\[
P(w | \alpha, H) = \frac{1}{Z_w(\alpha)} \exp(-\alpha E_w)
\]

………………………………(4.7)

Here, prior distribution is a Gaussian with a variance \( \sigma_w^2 = 1/\alpha \). Then, the objective function \( M(w) \) defined by Bayes’ theorem for a probabilistic model \( H \) becomes:

\[
P(w | D, \alpha, \beta, H) = \frac{P(D | w, \beta, H)P(w | \alpha, H)}{P(D | \alpha, \beta, H)} = \frac{1}{Z_M} \exp(-M(w))
\]

………(4.8)

Here \( M(w) \) is minimized to get the most probable vector \( w_{MP} \). Interpretation of the objective function \( M(w) \) as log probability has certain advantages in controlling the complexity of the model and calculation of the uncertainty in the model.

4.4 Advantages of Neural Networks with Bayesian Framework

Advantages of neural networks with Bayesian framework are listed below [62-64]:

(i) Automatic relevance determination, where the significance of the various inputs is quantified.

(ii) Uncertainty in the model predictions is quantified in terms of the error bars.

(iii) Model Comparison framework

(iv) Complexity of the model is controlled to prevent over-fitting.
4.4.1 Control of Complexity of the Model

Neural networks are highly non-linear models. Nonlinear models have a tendency to overfit data when their level complexity is increased. An overly complex model is highly sensitive to the variation in changes in input data, leading to poor generalization abilities. Generalization defines how well a model would predict an output when it was given unseen input data. Figure 4.3 shows the generalization ability as a function of model complexity [51]. The less complex model does not do a good job fitting the training data while the overly complex model fits the training data well, but does not fit the test data well. As the model complexity increases, the training error decreases continuously while test error decreases initially and then increases when the model began to over-fit. The test error measures the performance of the network. This trend is shown in Figure 4.4.

The Bayesian framework developed by Dr. MacKay treats the complexity in a natural manner. Important parameters that control complexity of the models are $\alpha$ and $\beta$. Control of these model parameters can be achieved through optimizing a quantity $P (\text{Data} \mid \text{Control Parameters})$. The detailed analysis of the optimization of these control parameters is given in [51, 65]. The important advantages related to Bayesian optimization of model control parameters are list below:

(i) Test set and validation set is not involved so all of the training data is utilized to model-fitting and model comparison.

(ii) Regularization constants, $\alpha$ and $\beta$ can be optimized in parallel with the optimization of ordinary model parameters.
(iii) The objective function in the Bayesian frame work is not noisy compared to a cross validation measure

(iv) Ability to optimize a large number of control parameters by evaluating the gradient of evidence with respect to control parameters

4.4.2 Automatic Relevance Determination

Bayesian framework has the ability to determine the relevance of each input. This framework considers the weight associated with each input as a separate class. Hyper parameter $\alpha_c$, a regularization constant associated with the weight of each input, can be seen as a measure of relevance of each input. Weights related to large values of $\alpha_c$ have a tendency to decay to zero, so the input associated with this weight is not a relevant one. The detailed description of the procedure of automatic relevance determination is described elsewhere [51].

4.4.3 Model Comparison

This framework [64] has model comparison built into it. This can be inferred by looking at the predictive distribution over different models:

$$P(H \mid D) \propto P(D \mid H)P(H)$$

It is difficult to know in advance how many hidden nodes will be required for particular sets of data. Each model uses different architectures, pre-processing and regularizers. So it is beneficial to compare solutions from different models to ensure a better fit.
4.5 Committee Models

Sometimes a single best model is not adequate to represent a set of data. It is not advised to rely solely on the measurements from a single model because each model gives importance to different variables, thus giving different predictions. So sometimes it is wise to take a prediction from a committee model, which is an average of different individual model predictions. Committee models do not change the complexity of the individual models. Averages of predictions and error bars from a committee model are defined below.

Predictions from Committee Model: $\bar{y} = \frac{1}{L} \sum_{l} y_l^{(i)} \quad \ldots \ldots \ldots \ldots \ldots (4.9)$

Error Bars from Committee Model: $\sigma^2 = \frac{1}{L} \sum_{l} \sigma_{y}^{(i)^2} + \frac{1}{L} \sum_{l} \left(y_{l}^{(i)^2} \right) + \frac{1}{L} \left(y_{l}^{(i)} - \bar{y}\right)^2 \quad \ldots (4.10)$

Using the Bayesian inference we addressed uncertainty in the model predictions in terms of error bars, automatic control of complexity of the neural network model and automatic relevance determination of the different inputs. The following chapters use neural networks to predict mechanical properties such as yield strength, tensile strength and fracture toughness in $\alpha+\beta$ processed and $\beta$ annealed $\alpha+\beta$ Ti alloys. Based on the model predictions we can also design control experiments to bring out the mechanisms.
Figure 4.1 Schematic of the three layer neural network

Figure 4.2 Uncertainty in model prediction, region A, noise in data and region B, data lean region [41]
Figure 4.3 Effect of model complexity on generalization ability (a) poor generalization, (b) good generalization, and (c) over-fitting [51]
Figure 4.4 Errors as a function of complexity of the model [41]
Chapter 5 Neural Network Modeling of Tensile Properties of $\alpha + \beta$ Processed Titanium Alloys

5.1 Abstract

Although the properties of structural alloys such as $\alpha + \beta$ processed Ti-6Al-4V are strongly dependent upon their microstructure, modeling of such properties is challenging because the alloys’ microstructure varies in a complex fashion. Predicting properties becomes significantly more difficult when composition is added as an additional degree of freedom. This chapter describes the application of a rules-based approach, a neural network with a Bayesian framework as a solution to property modeling and includes the population of a database containing information regarding alloy composition, microstructure, and tensile properties for a set of $\alpha + \beta$ processed $\alpha/\beta$ Ti-alloys based on Ti-$x$Al-$y$V ($4.76 < x < 6.55$; $3.30 < y < 4.45$) with controlled variations in interstitial O and Fe ($0.07 < \text{wt}\% \text{O} < 0.20$; $0.11 < \text{wt}\% \text{Fe} < 0.41$). The developed models have been used to study the effects of individual alloying elements and microstructural variables on tensile properties. For tensile properties, the Al and O are important composition variables that significantly strengthen the alloy, while the volume fraction of the equiaxed alpha ($\alpha_p$) is the most important microstructural variable.
5.2 Introduction

The attractive combination of properties present in many Ti alloys has led to extensive research and the subsequent application of these alloys across a wide range of industries (e.g., aerospace, automotive, biomedical) [18, 66, 67]. Interestingly, many of these structural alloys, including the commonly used α/β alloy Ti-6Al-4V, can exhibit an extended range of tensile properties (e.g., 725-930MPa). Researchers have claimed that, for a specific Ti alloy, the mechanical properties are dependent upon the microstructure [23, 68-71]. Therefore, any predictive model for the provision of mechanical properties that is developed should, at the very least, relate microstructure with properties. However, until recently, the best known microstructure-property relationships, for the case of α/β Ti alloys, have been qualitative rather than quantitative in nature [20, 21, 71-73], and often limited to specific alloy systems. The prediction of properties for a particular microstructure involves the declaration of an outcome (i.e., a property), based on an observation or scientific reason (i.e., the material microstructure) prior to a true measurement (e.g., a tensile test). Although, significant research efforts are aimed at the development of mechanistically based predictive models, such models that have long development cycles and are still in their infancy. Therefore, the current chapter focuses on the development of a predictive model based on a short-term approach, i.e., rules based, such as neural networks [61, 62, 64, 74]. The successful application of such neural networks has been described by researchers who have developed predictive models and used them to quantitatively describe the dependencies of properties upon microstructure.
While these models have proven to be the most accurate tools developed for the prediction of mechanical properties, each has been developed for a specific alloy composition (e.g., Ti-6.4Al-4V-0.16Fe-0.18O – the composition from a single melt of a typical Ti-6-4 alloy). Thus, there exists a significant risk in the application of such models to alloys that do not fall within a small region of the compositional and microstructural space. Since these models are based upon heat treatments designed to provide an “equilibrium” microstructure (e.g., very little variation in the ratio of the two equilibrium phases), and do not currently allow for any variation in composition, there is no direct or indirect inclusion of non-microstructural feature based strengthening mechanisms, such as solid solution strengthening, in the models.

Given the absence of a robust, predictive model relating composition, microstructure, and mechanical properties, many uncertainties exist regarding the prediction of mechanical properties. There is not a tool to predict a property minimum (of critical importance to designers [19]) given a particular microstructure from a specific heat (i.e., unique alloy composition) or a computational tool to inform industrial designers what heat treatment (i.e., microstructure) is required to achieve a minimum design allowable given a slight change in alloy composition. Indeed, the specifications for Ti-6Al-4V exhibit an extended composition range (see Table 5.1), resulting in average minimum properties that can vary by more than 20%.

Therefore, there are three principle aims of the research that is presented in this chapter. The first is to develop a well-populated database for a α+β processed Ti-based alloy with significant variations in both composition and microstructure. Such variations
have been achieved using standard industrial practices. The composition space is the Ti-Al-V system, nominally around the alloy Ti-6-4, with explicitly designed variations in not only the Al and V, but also the Fe and O interstitial content. The second is to use the database to develop Bayesian neural network models for the prediction of properties given a specific composition and microstructure. The third is for a first approximation of functional dependencies of the two types of variables (composition and microstructure) on properties. These functional dependencies can be used in many ways, including property prediction, identification of missing microstructural features or the determination of fundamental mechanisms for subsequent inclusion in mechanistic-based models. This research has several important aspects that differ from similar, previous work [20]. The most important difference is that the alloys were prepared and processed using standard, industrially accepted methods; subsequent thermomechanical processing was also performed using standard methods. The other differences include the size and degree of sophistication of the resulting database (e.g., this study has more samples from a smaller region of compositional space), and the fact that the alloys of interest are α+β processed rather than β processed. It is interesting to note that the magnitude of the influence of composition found in this chapter is very similar to that found in the previous work [20] even though the processing routes were different.

5.3 Experimental Procedures

A total of nine different Ti alloys (based around Ti-6Al-4V) were produced with deliberate variations in the relative amounts of the individual alloying elements, including the interstitial Fe and O contents. The ranges of alloy variations can be found in Table
5.2 and Table 5.3, as measured by Timet using ICP, with reference to two of the standards for the composition of the alloy Ti-6Al-4V. The Al content was allowed to range well below that of standard Ti-6Al-4V, while the V was allowed a slightly extended range. The Al concentration was intentionally kept below the maximum allowed by the standards (see Table 5.1 and Table 5.3) in order to avoid the onset of short-range ordering of the HCP $\alpha$ phase that would result in embrittling of $\alpha_2$ domains. The O level had a slightly extended range, notably with an alloy that was extremely O lean (0.07 wt% - 700 ppm). The Fe level had a significantly extended range. For each of the nine alloy compositions, six samples were exposed to different thermomechanical processing histories, producing a total of 54 samples. Figure 5.1 (a-d) shows three-dimensional scatter plots illustrating the distribution of the 54 samples across the compositional space. As can be seen, there is still a large fraction of composition space that remains unrepresented as discrete points in the dataset. This is notable for two reasons. First, it underscores the ability of the neural networks to make a true prediction, rather than a data-mined estimate, of the property. Second, it illustrates the effectiveness of the Bayesian neural network in handling the missing data or data-lean regions of input space. The consequences of such data-lean regions will be discussed. It should be noted that all samples were taken from the same radius of a round billet, thus minimizing the differences in strain during deformation and thus the resulting differences in texture.

The samples were subjected to room temperature tensile tests. Following the tensile tests, an undeformed section of the grip from each sample was sectioned for metallographic preparation. Following traditional metallographic techniques, the samples
were characterized using a FEI/Philips Sirion scanning electron microscope (SEM) operating in backscattered mode at 15 kV with a resolution of approximately 3.0 nm. Each image had a resolution of 3872 x 2904 pixels and a bit depth of 8. The high resolution afforded using the SEM, especially when compared with other available techniques (e.g., optical micrographs), is of paramount importance because it is critical to provide the highest possible fidelity quantified microstructural data to the neural networks. The specimens were imaged at four random locations to avoid overlap and edge effects. Microstructural features present in the four micrographs were quantified using the stereological techniques [59]. These features include the size of the equiaxed alpha (equiaxed-α size, µm), the volume fraction of equiaxed alpha ($F_{V_{equiaxed-α}}$), the volume fraction of total alpha ($F_{V_{total-α}}$), and the width of the alpha laths in the transformed β regions. The width of the α lamellae was measured using Gundersen’s derivation (α-lath width, µm) [75].

The experimentally determined data regarding alloy composition, microstructure, and tensile properties was included in a database for additional analysis. The database was analyzed using an artificial neural network with Bayesian architecture, after the work of D. Mackay [63]. This probabilistic neural network was used to develop a model that predicts the interrelationships between microstructure and tensile properties of the α+β processed Ti-6Al-4V alloy. Once developed, the neural network can be used in one of two ways. The first is involved the development of a predictive tool, where the properties of a test dataset are predicted. The test dataset represents a fraction of the 54 samples that were not used for the training, in this case, 38 samples were used to train the model and
16 were used to test the model. The second is the use of the neural network to perform virtual experiments. These are usually control experiments where the values of given microstructural features can be set to, or held at, given values. In this way, it is possible to obtain information that is not readily available from actual experiments where such control of individual microstructural features is not possible. The ability to perform virtual experiments is very advantageous; it can be used to determine functional dependencies and identify basic mechanisms, missing data, and significant missing input parameters, as described elsewhere [76]. This paper will describe not only the functional dependencies, but also the identification of significant missing input parameters and missing data.

5.4 Results and Discussion

5.4.1 Database Development

Figure 5.2 (a-d) shows backscattered electron micrographs of representative samples used in the population of the database. There is a distinct difference in the size and volume fraction of the equiaxed α particles, as well as a difference in the thickness of the Widmanstätten α-laths. The total range in mechanical properties and average microstructural variables can be seen in Table 5.4 and Table 5.5, respectively. It is interesting to compare the range of properties produced with some of the standard ranges for Ti-6Al-4V. The minimum UTS and YS produced in this study were far below the property specifications of similarly processed Ti-6Al-4V, although the maximum values of properties in this study were less than the maximum expected for Ti-6Al-4V. One
might speculate that this is related to the alloy compositions, particularly the Al composition, which was kept below the minimum for the alloy Ti-6-4, based on the standards given in Table 5.1.

Figures 5.3 (a-d) shows four microstructures, which have nominally the same yield strength (~838 MPa). The total range of yield strength within these four microstructures is 15 MPa, or approximately 5% of the total property range. However, both the microstructures and the alloy compositions vary significantly. Such variation illustrates the importance in establishing the databases for subsequent analysis using tools such as neural networks. Figure 5.3 also underscores the complex interrelationships that exist between alloy composition and microstructure, and the effect of such interrelationships on the resulting properties. In this case, the total effect on properties resulting from the collective (and offsetting) changes in composition and the size and distribution of microstructural features is negligible.

The coefficient of variation (CV) of particular microstructural features present in the database is similar to those found in previously published work on the quantification of α+β processed microstructures. The CV measures of dispersion of the microstructural data in the data series around the mean and is defined as the ratio of the standard deviation to the mean. The average CV for the volume fraction of equiaxed α was the worst (~6.2%) while the CV for the volume fraction total α was the best (0.8%). The average CV for the size of the equiaxed α particles and the thickness of the Widmanstätten α-laths were 4.4% and 6.0% respectively. In general, the average CV indicates that the database contains measurements of high fidelity.
5.4.2 Overall Model Accuracy

In order to determine the importance of the inclusion of alloy composition on the quality of the neural network models, two sets of Bayesian neural network models were developed. The first set of models (yield strength and ultimate tensile strength) was based solely on the four microstructural inputs (equiaxed alpha size, the volume fraction of equiaxed alpha, the volume fraction of total alpha, and the width of the \( \alpha \) lamellae in the transformed \( \beta \) regions). The second set of models was based on an expanded set of eight inputs, including four compositional inputs (Al, V, O and Fe in wt\%) in addition to the four microstructural inputs. These models were then evaluated to determine their quality (e.g., their accuracy). One method of analyzing the quality of each model is by comparing the experimentally measured properties with those predicted by the model. Figure 5.4 (a-b) and Figure 5.5 (a-b) show the predicted vs. experimental tensile properties (yield and ultimate tensile strengths) for the two types of models (i.e., with and without composition as an input variable). In each graph, the sloped lines provide a visual guide for a predicted value that is \( \pm 5\% \) of the experimental value. Included within these plots are both training and testing datasets. The model is ranked based on the minimum error in test set. For a particular model, the test set consists of microstructural and compositional data contained \textit{within the range} of the training database, however the model does not use this data in the training stage.

As can be seen from Figure 5.4 (a-b) and Figure 5.5 (a-b), when composition is excluded as an input variable, the model quality is poor, with errors in prediction of both the training and test dataset in excess of 10\% of the experimental value. However, when
composition is included as an input variable, as shown in Figure 5.4 (b) and Figure 5.5 (c), the results are significantly improved. The model predictions for both the training and test datasets are within ± 2.5%. In addition to these figures, which provide a qualitative analysis of the models, it is possible to determine the figures of merit for the accuracy of the model. Such figures of merit include the comparison of the average and maximum deviations between the experimentally measured and neural network predicted properties using the both the training and testing datasets (δ_{Avg} and δ_{max}), the average and maximum uncertainty (i.e., error) predicted by the Bayesian model (E_{avg} and E_{max}), and the number of cases where the experimentally measured and predicted properties are statistically equivalent, given the predicted uncertainty of the data point. The deviations (δ) and errors (E) are normalized with respect to the experimental and predicted values, respectively, and expressed as a percentage. The values for the deviations and errors are shown in Table 5.6. For the training dataset, 17/40 data points had statistically equivalent yield strengths and 23/40 had statistically equivalent ultimate tensile strengths. Likewise, for the testing dataset, 5/16 had both statistically equivalent yield and ultimate tensile strengths.

The significant improvement in the model quality upon the addition of overall alloy composition strongly suggests that, in addition to affecting the microstructural variables, the overall composition has a marked impact on the properties through mechanisms related to phase composition, possibly including solid solution strengthening as well as modifications of solution distribution and interfacial structures.
5.4.3 Mono-variable Functional Dependencies

One of the applications of Bayesian neural networks is the use of the developed models to determine the influence of individual microstructural features on given properties, (i.e., functional dependencies). For example, in Figure 5.6 (a), the variation of yield stress as a function of the average $\alpha$ lath thickness is plotted directly from the database. Similarly, in Figure 5.6 (b), the variation in yield stress as a function of the wt% O in the alloy is plotted directly from the database. As can be seen, the resulting points are somewhat scattered, with a generally increasing trend in strength as values for both the average $\alpha$-lath thickness and wt% O increase. The scatter in the data is the result of simultaneous variations in the values of the other microstructural and compositional features represented in these samples. It is necessary to perform a set of controlled experiments where the value of a given feature, in this case the width of $\alpha$ laths, is varied systematically as the values of all other features are set to certain constant values, often their averages. Such controlled experiments cannot be done experimentally. Indeed, it may not be possible to physically manipulate the microstructure in this manner as the microstructural features are interrelated. However, these experiments can be performed computationally using the developed neural network model. Thus, using the model which includes both compositional and microstructural information, all but one of the microstructural and compositional features are set to their average values while the value of a selected input is systematically varied (i.e., mono-variable). In this manner, a reasonable representation of the resulting variation in the predicted yield stress for a given input is provided. In the case of these virtual experiments, the functional
dependencies are unambiguous. The use of virtual experiments to determine the role of microstructure on properties was first done by Grylls [77] in the case of marine alloys.

Figures 5.7 (a-d) show the functional dependencies of the four compositional variables while Figure 5.8 (a-d) shows the functional dependencies of the four microstructural variables. Similarly, Table 5.7 includes a measure of the degree to which a particular input variable affects the mechanical properties when all other features are set to their average values, given a particular unit of measure (i.e., percentage or micron). Although Table 5.7 indicates that two microstructural features related to size (i.e., the mean equiaxed alpha particle size and the average thickness of the Widmanstätten alpha laths) influence the mechanical properties to a greater extent, the volume fraction of equiaxed alpha particles is the microstructural feature that has the greatest influence on the resulting tensile properties (see Figure 5.8 (a)). This is largely due to the extended range in volume fraction of equiaxed alpha that can be seen in these microstructures, resulting in a variation in yield strength of almost 60 MPa. Thus, not only it is important to consider the normalized influence of a particular variable on the properties, but also the extent of the range that a particular variable can exhibit. For example, for two microstructural features related to scale, it is also important to note that while the thickness of the Widmanstätten alpha laths may yield a more significant negative influence on properties per micron increase in thickness, the range of lath thickness that can be affected by thermomechanical processing is smaller than the range of equiaxed alpha particle sizes, ~0.345 < t_{lath} < 0.667 and 4.79 < d_{equiaxed} < 8.39, respectively. The general trends shown in Figure 5.8 are not surprising. Indeed, the trends in volume fraction of
total alpha and Widmanstätten alpha lath thickness are similar to what has been previously shown. Likewise, it is not surprising that as the size of the equiaxed alpha particles decreases, the strength would increase. This trend in volume fraction equiaxed alpha most likely results from the difference in size of the microstructural features being considered. That is, one might speculate that the early dislocation activity may be more easily accommodated in the equiaxed alpha particles rather than the Widmanstätten alpha laths, and that a higher fraction of equiaxed alpha would result in a lowering of the yield strength. The size of equiaxed of alpha can be related to slip length, which increases with increase in the size of the equiaxed alpha. The increase in slip length causes a decrease in yield strength. The reduction in yield strength due to increase in volume fraction of the equiaxed alpha can also be attributed to solute partitioning effect in which alpha stabilizers, Al and O partitioned into equiaxed alpha particles causing a reduction in basic strength of Widmanstätten alpha in a transformed beta matrix.

A similar analysis of the dependency of properties on alloy composition yields stronger influences, particularly with respect to the \( \alpha \)-stabilizing elements, Al and O. In addition to showing the influence of each alloying element on the yield and ultimate tensile stresses, the total possible change in yield strength has been calculated for the Mil. Spec. F-83142A-#6, based on the minimum and maximum amount of each alloying addition (see Table 5.7). For the Fe and O measurements, the calculations are made for the increase in strength of a non-extra low interstitial (ELI) grade alloy with respect to an ELI grade, given the maximum ELI impurities. These calculations are made strictly as a function of composition with all other microstructural variables held constant. The Al
and O can each result in an increase in the yield stress of the material of 63.6 and 66.0 MPa (per wt%), respectively. Based on this model, the increases in strength afforded by changes in composition are more significant than the changes in strength offered by modifications to the microstructure.

5.4.4 Bi-variable Functional Dependencies

In a fashion similar to the determination of functional dependencies as described above, it is possible to use the Bayesian neural network models to extract synergistic functional dependencies by setting all but two of the microstructural and compositional features to their average values while the values of the selected inputs are systematically varied (i.e., bi-variable). In this case, Figures 5.1(a-d) may be considered analogous to Figure 5.6 (a-b), where the experimentally measured property values present in the database are plotted against two of their input variables, in this case their compositions. As was the case for Figure 5.6 (a-b), it is important to recall that each of the other input variables, both microstructural and compositional, have variations that are not captured in such a graphical representation. However, as was the case for the mono-variable functional dependencies, when the remaining variables are fixed at an experimental value, one can investigate synergistic influences present in bi-variable functional dependency plots. For example, it is possible to hold the microstructural variables constant, and vary both Al and V with a fixed interstitial content to produce a three-dimensional estimation of a property (e.g., yield strength) with simultaneous variations in both Al and V, such as those shown in Figure 5.9 (a-b). The exercising of the Bayesian
neural network models in this fashion represents a new method of investigating synergistic effects on the functional dependencies.

The Bayesian neural network has been used to produce a bi-variable functional dependency (Al and V) to show the influence that the various alloying elements have on the yield strength (Figure 5.9 (a)) and ultimate tensile strength (Figure 5.9 (b)). As can be seen in these figures, there are three sets of predictions corresponding to three different sets of Fe and O interstitial levels, each represented as a surface. As expected, as the concentration of the interstitial alloying elements increases, the overall strength of the alloy increases significantly. Similarly, these bi-variable functional dependencies also capture the very strong influence of Al on both the yield and ultimate tensile strengths. However, there is an interesting result with respect to the influence of V on the properties. Such an influence is the result of a missing parameter, as described below.

In a similar fashion, the Bayesian neural networks can be used to investigate bi-variable functional dependencies where one variable is related to microstructure and the other to composition. Figure 5.10 (a-b) shows the original data and bi-variable functional dependencies for yield strength as a function of equiaxed α particle size and wt% Al. Included are three different levels of oxygen, each represented as a two-dimensional surface.

5.4.5 Identifications of Missing Parameters

Although these predictive models are of the highest fidelity and provide the most accurate predictions of properties for Ti-based alloys (e.g., within ± 2.5%) currently
available, there are microstructural parameters that are likely missing as inputs to the models. Recall the improvement in the overall quality of the models, based on the errors, upon the inclusion of composition - a set of variables that significantly influences the overall tensile properties. In a similar fashion, it would be reasonable to expect the presence of an error, albeit small, to be the result of missing parameters and/or the fidelity of the data. Although these models do capture the most recognizable microstructural features (e.g., the volume fraction and size of equiaxed \( \alpha \) particles), they do not contain all possible microstructural features. As an additional complication some missing microstructural features, such as the formation of nanometer-scaled secondary (or tertiary) alpha, might only be present in a small fraction of samples with a certain alloy composition and heat treatment.

As described above, the presence of missing microstructural features is certainly easily observed when analyzing the quality of a model. The errors become large when the important microstructural features are left out in the training of the networks. However, the presence of selected missing features may also be identified by comparing virtual experiments (e.g., the functional dependencies) to determine whether the missing feature is indirectly related to input that is being varied. Consider Figure 5.9 (a), which has a variation in the influence of the V on the predicted yield strength for different interstitial contents. For alloys with low interstitial levels, the influence of V on the yield strength is positive. However, for alloys with high interstitial levels, the influence of V is negative. Likewise, a similar variation in the bi-variable ultimate tensile strength functional dependency is observed, although it is less obvious. In this case, the interstitial level
appears to influence the curvature of the two-dimensional surface. As fundamental strengthening mechanisms related directly to composition (e.g., solid solution effects) are not expected to change their influence over the same compositional regime (i.e., the same functional dependencies are expected for different interstitial levels), it is clear that something is adversely affecting the model, even if only minimally. It might be useful to determine any synergistic effects among existing inputs while trying to identify any missing parameters. For example, the V-Fe and V-O interactions were probed. Although it is tempting to suspect a synergistic V-Fe interaction (e.g., both β-stabilizers), it is actually a synergistic V-O effect (see Figure 5.11 (a-b)) that is related to the missing input variable. However, this still does not identify potential missing microstructural data. A careful reexamination of the microstructures indicates small, unquantified differences between samples with nominally the same average microstructural features, Al content, and a variation in the interstitial levels, V levels, and a resulting difference in properties.

Table 5.8 gives three sets of experimental data with nearly identical quantified microstructural features. As can be seen, while the Al and O are nearly identical within a given set, both the V and Fe levels are different. Although the nature of the dataset made it necessary to consider two variables (V and Fe) simultaneously, the significant synergistic V-O effect was excluded by holding O nearly constant. Additionally, it is noted that while an increase in the Fe is expected to result in an increase in the yield strength, the magnitude of the increase in yield strength predicted by the model further indicates that there are additional, missing factors. Indeed, the modest increase in yield strength caused by a small change in Fe (<20 MPa) cannot explain the total difference in
yield strengths for samples whose quantified microstructural parameters are nearly identical. However this change is important because strengthening due to Fe coming from the β-phase, which is on average only 15%.

For cases where the O content is high (e.g., ~0.2 wt% or 2000 ppm) and the Al content is low, the data indicates that the sample with lower V and higher Fe has higher yield strength. Figure 5.12 shows a set of mono-variable functional dependencies of V on the yield strength for two Fe levels, given the average microstructure, Al, and O levels of the two samples. As can be seen, the sample with higher V has lower yield strength, thus accurately representing the variations in these two samples, and is consistent with Figure 5.9. Figure 5.13 (a-b) shows SEM micrographs of the two samples. While the overall microstructures appears to be very similar, the microstructure present in Figure 5.13 (b) (the alloy with low V and the higher yield strength) does have several instances of small fractions of well-developed nanometer-scaled secondary (or tertiary) alpha. Additionally, there are differences in the thickness and continuity of the β-ribs.

For cases where the O content is low (e.g., ~0.075 wt% or 750 ppm) and the Al content is low, the data indicates that the sample with higher V and higher Fe has higher yield strength. Figure 5.14 shows a set of mono-variable functional dependencies of V on the yield strength for two Fe levels, given average microstructure, and average Al and O levels. As can be seen, the higher V results in higher yield strength, thus accurately representing the variations in these two samples, and is consistent with Figure 5.9 (a). Figure 5.15 (a-d) shows SEM micrographs of the two samples. For this case, it is clear that the aspect ratio of the equiaxed alpha particles is different, thus representing a
missing microstructural parameter. In addition to this missing microstructural parameter, related to the unknown, true three-dimensional shape of the equiaxed alpha particles, the microstructure present in Figure 5.15 (b-c) (the alloy with high V and the higher yield strength) also has several cases where there is a small fraction of well-developed nanometer-scaled secondary (or tertiary) alpha. Thus, there is consistency between these two cases, which indicates that nanometer-scaled alpha appears to be a variable that increases the strength.

For cases where the O content is low (e.g., ~0.075 wt% or 750 ppm) and the Al content is high, the data indicates that the sample with higher V and lower Fe has higher yield strength, although not significantly higher. This is an important case because whatever strengthening mechanism is present has offset the positive increase of the higher Fe content. Figure 5.16 shows a set of mono-variable functional dependencies of V on the yield strength for two Fe levels, given the average microstructure, Al and O levels. As can be seen, the higher V results in higher yield strength, thus accurately representing the variations in these two samples, and is consistent with Figure 5.9 (a). Figure 5.17 (a-d) shows SEM micrographs of the two samples. While the overall microstructure appears to be very similar, including the presence of nanometer-scaled alpha, there are two subtle differences. The first is the fraction of the well-developed nanometer-scaled alpha, which is higher for the high V and higher strength alloy shown in Figure 5.17 (a-b). The second is that the transformed β phase has more of a basketweave appearance in Figure 5.17 (c-d).
The presence of such fine scale alpha laths appears to be associated with the higher yield strength for all three cases, but interestingly appears to be stable at different V levels for different interstitial O contents. This is perhaps the origin of the synergistic V-O influence. Clearly, the presence of such fine scale alpha laths is a missing microstructural variable that may be contributing to the overall strength of the alloys. However, even with such a variable missing, the model still works to predict the properties to within ± 2.5%. This is likely the result of the indirect relation of the presence of such fine scale alpha to the overall alloy composition, particularly the V and O contents. Thus, although the presence of fine scale alpha laths was not an independent variable in the model, it was indirectly present through the complicated synergistic V-O influence. Clearly an analysis of unexpected synergistic effects will help identify missing microstructural parameters.

5.4.6 Exercising the Model to Identify Data – Lean regions

The incorporation of uncertainty in a neural network model is a benefit associated with the use of Bayesian neural networks. This uncertainty is represented in terms of error bars. The error bars calculated for the predicted values become large in areas where data is noisy or data is sparse. The error bars are minimized in areas where data is rich. Were the database to be extended, it would be beneficial to target samples within such data-lean regions, as this would improve the models.

An example of the error associated with a bi-variable functional dependency of yield stress is shown in Figure 5.18. Here, it is clear that there is less data at high V
levels (6 samples at ~4.5 wt%, V) compared with low V levels (24 samples ~3.4 wt%, V). A comparison of the errors on this graph (~3-7 MPa) to the maximum errors in the predictions of yield strength and UTS (~8-10 MPa) further demonstrates the self-consistency of these models.

5.4.7 Other Novel Uses

In addition to using the Bayesian neural network to predict properties and identify microstructural features and compositional variables that significantly influence the mechanical properties, it can be potentially exploited in a variety of novel ways to tailor the microstructure or composition to affect a certain property. As discussed in the introduction, the ability to tailor the composition or microstructure to ensure that the average microstructure is within a specific allowable range would be beneficial to many industries. Therefore, one novel use of the model is the development of n-dimensional isoyield contours (n ≤ number of input variables), where such contours might, for example, indicate these minimum design allowances. For example, in Figure 5.9 (a), there is a three-dimensional isocontour indicating the combination of Al, V, and interstitial levels that result in yield strength of at least 880 MPa, given an average microstructure. This is useful in alloy grade selection; for example if a particular yield strength (e.g., 880 MPa) is required for a non ELI grade Ti-6-4 with a particular microstructure (e.g., an average microstructure established by the database), it would be possible to evaluate the chemical certification with respect to the combination of Al and V. For example, the properties would not be met for that given microstructure if the chemical certification indicated either enrichment in V or depletion in Al, resulting in an
average composition to the right of the line indicating 880 MPa in Figure 5.9 (a). However, either depletion in V or enrichment in Al would satisfy the design criterion. While such an example is illustrative, consider Figure 5.19, a plot that combines three of the most significant variables that influence the yield strength, the wt% Al, wt% O and equiaxed alpha particle size. The surface presented on this graph represents the combination of the three variables (all others held at an average value) that result in a yield strength of 830 MPa. The volume under the surface has a yield strength that exceeds 830 MPa, while the volume above the surface had a yield strength that is less than 830 MPa. Therefore, there is an inherent degree of guidance in microstructural development for alloys that might not satisfy specifications by a marginal amount. It is interesting to note that there are certain conditions where it is impossible to produce an alloy whose yield strength is 830 MPa, particularly with alloys lean in Al or O.

Another novel use is the exploitation of such results to guide in the determination of fundamental mechanisms that control physical properties. For example, while it is possible to speculate about the relationship of an increase in strength to an associated increase in O or Al (e.g., through a modification of the Peierls stress) or an associated decrease in the volume fraction equiaxed alpha particles (described previously), models such as this do not necessarily identify fundamental strengthening mechanisms. However, they can be used to jumpstart new avenues of research focused on identifying the most important fundamental strengthening mechanisms. For example, based on these results, there is ongoing research to identify the critical resolved shear stress for the different slip systems in α-Ti as a function of local Al and O composition and orientation.
Similarly, there is evidence that the presence of nanometer-scaled alpha and thickness of the \( \beta \) ribs play a role in strengthening.

### 5.5 Conclusions

This chapter has demonstrated the application of a Bayesian neural network to develop the most accurate predictive models for the stipulation of properties in \( \alpha+\beta \) processed Ti-6-4 alloys to date. In addition to the development of a well-populated database with both microstructural and compositional variables for the training of the neural network, this research has presented results of the neural network such as the predictions of tensile properties, functional dependencies and synergistic effects observed in bi-variable functional dependencies. For these alloys, the Al and O levels are the most important compositional variables that influence strength. The volume fraction of the equiaxed \( \alpha \) particles is the most important microstructural variable. The use of these models has resulted in the determination of a potential V-O synergistic interaction that may lead to the formation of nanometer-scaled \( \alpha \) laths under certain conditions, resulting in an increase in the yield strength. Finally, future uses of the network were discussed. These included using the network to guide alloy selection, to determine microstructural requirements, and to guide further fundamental research.
### Table 5.1 Different specifications for composition of Ti-6Al-4V alloy

<table>
<thead>
<tr>
<th>Specification</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>O (max)</th>
<th>N (max)</th>
<th>Fe (max)</th>
<th>C (max)</th>
<th>H (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AMS ELI Plate Specification 4095</td>
<td>Balance</td>
<td>5.6-6.3</td>
<td>3.6-4.4</td>
<td>0.12</td>
<td>0.03</td>
<td>0.25</td>
<td>0.05</td>
<td>125 max</td>
</tr>
<tr>
<td>AMS Frg. Ann. Specification 4920</td>
<td>Balance</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>0.20</td>
<td>0.05</td>
<td>0.30</td>
<td>0.10</td>
<td>125 max</td>
</tr>
<tr>
<td>ASTM Frg. Ann. Specification B381</td>
<td>Balance</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>0.20</td>
<td>0.05</td>
<td>0.40</td>
<td>0.10</td>
<td>125 max</td>
</tr>
<tr>
<td>MIL Frg. Ann. Specification F-83142A-#6</td>
<td>Balance</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>0.20</td>
<td>0.05</td>
<td>0.30</td>
<td>0.08</td>
<td>150 max</td>
</tr>
<tr>
<td>MIL Frg. Ann. ELI Specification F-83142A-#6</td>
<td>Balance</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>0.13</td>
<td>0.05</td>
<td>0.25</td>
<td>0.08</td>
<td>125 max</td>
</tr>
<tr>
<td>MIL Sharp Strip Specification T-9046A</td>
<td>Balance</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>0.20</td>
<td>0.05</td>
<td>0.30</td>
<td>0.08</td>
<td>150 max</td>
</tr>
</tbody>
</table>

### Table 5.2 Range in alloying elements in Ti-6Al-4V and in the current database

<table>
<thead>
<tr>
<th>Specification</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>O (max)</th>
<th>Fe (max)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIL Frg. Ann. ELI Specification F-83142A-#6</td>
<td>Balance</td>
<td>5.5-6.5</td>
<td>3.5-4.5</td>
<td>0.13</td>
<td>0.25</td>
</tr>
<tr>
<td>AMS Frg. Ann. Specification 4920</td>
<td>Balance</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>0.20</td>
<td>0.30</td>
</tr>
<tr>
<td>Current Extended Compositional Dataset</td>
<td>Balance</td>
<td>4.76-6.55</td>
<td>3.30-4.45</td>
<td>0.07-0.20</td>
<td>0.11-0.41</td>
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</table>
Table 5.3 Chemical analyses of nine alloys in the database

<table>
<thead>
<tr>
<th></th>
<th>Aluminum (wt%)</th>
<th>Vanadium (wt%)</th>
<th>Iron (wt%)</th>
<th>Oxygen (wt%)</th>
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<tr>
<td>1</td>
<td>5.640</td>
<td>3.830</td>
<td>0.252</td>
<td>0.135</td>
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<tr>
<td>2</td>
<td>4.766</td>
<td>3.297</td>
<td>0.110</td>
<td>0.080</td>
</tr>
<tr>
<td>3</td>
<td>6.514</td>
<td>4.290</td>
<td>0.110</td>
<td>0.079</td>
</tr>
<tr>
<td>4</td>
<td>6.495</td>
<td>3.313</td>
<td>0.107</td>
<td>0.190</td>
</tr>
<tr>
<td>5</td>
<td>4.790</td>
<td>3.350</td>
<td>0.390</td>
<td>0.196</td>
</tr>
<tr>
<td>6</td>
<td>4.850</td>
<td>4.450</td>
<td>0.110</td>
<td>0.198</td>
</tr>
<tr>
<td>7</td>
<td>4.760</td>
<td>4.270</td>
<td>0.390</td>
<td>0.072</td>
</tr>
<tr>
<td>8</td>
<td>6.550</td>
<td>4.380</td>
<td>0.407</td>
<td>0.197</td>
</tr>
<tr>
<td>9</td>
<td>6.550</td>
<td>3.380</td>
<td>0.394</td>
<td>0.071</td>
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</table>

Table 5.4 Tensile property range in database

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<tr>
<th>Minimum Property</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
<th>Elong. (%)</th>
<th>RA (%)</th>
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<tbody>
<tr>
<td>Maximum Property</td>
<td>747</td>
<td>683</td>
<td>14.1</td>
<td>26.2</td>
</tr>
<tr>
<td>Average Property</td>
<td>1036</td>
<td>955</td>
<td>20.4</td>
<td>52.3</td>
</tr>
<tr>
<td></td>
<td>888</td>
<td>818</td>
<td>17.8</td>
<td>42.6</td>
</tr>
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</table>
Table 5.5 Ranges of experimentally quantified microstructural parameters in a tensile database

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<tr>
<th></th>
<th>Equiaxed α size (μm)</th>
<th>Fv equiaxed α (%)</th>
<th>Fv total α (%)</th>
<th>Widmanstätten α-lath size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum</td>
<td>4.79</td>
<td>32.1</td>
<td>85.3</td>
<td>0.345</td>
</tr>
<tr>
<td>Maximum</td>
<td>8.42</td>
<td>81.5</td>
<td>93</td>
<td>0.67</td>
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<tr>
<td>Average</td>
<td>6.91</td>
<td>55.5</td>
<td>90.3</td>
<td>0.522</td>
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Table 5.6 Errors assessing the model quality in developed neural network models

Table 5.7 Effect of composition and microstructural features on tensile properties calculated from virtual experiments
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<th>Case 1:</th>
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<tbody>
<tr>
<td></td>
<td>Al</td>
<td>V</td>
<td>O</td>
<td>Fe</td>
<td>Equiaxed α size (µm)</td>
<td>Fv equiaxed α (%)</td>
<td>Fv total α (%)</td>
<td>Widmanstätten α-lath size (µm)</td>
<td>UTS (MPa)</td>
<td>YS (MPa)</td>
</tr>
<tr>
<td>Sample A</td>
<td>4.85</td>
<td>4.45</td>
<td>0.196</td>
<td>0.11</td>
<td>7.09</td>
<td>58.8</td>
<td>91.0</td>
<td>0.612</td>
<td>889.4</td>
<td>809.4</td>
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<tr>
<td>Sample B</td>
<td>4.79</td>
<td>3.35</td>
<td>0.196</td>
<td>0.39</td>
<td>7.06</td>
<td>64.6</td>
<td>91.6</td>
<td>0.670</td>
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<tr>
<td>Average</td>
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<td>3.50</td>
<td>0.20</td>
<td>0.20</td>
<td>7.07</td>
<td>61.7</td>
<td>91.3</td>
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<tr>
<td>Sample A</td>
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<td>4.27</td>
<td>0.07</td>
<td>0.39</td>
<td>5.84</td>
<td>54.3</td>
<td>89.5</td>
<td>0.475</td>
<td>796.3</td>
<td>731.5</td>
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<tr>
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<td>4.77</td>
<td>3.30</td>
<td>0.08</td>
<td>0.11</td>
<td>6.01</td>
<td>51.7</td>
<td>92.0</td>
<td>0.505</td>
<td>757.0</td>
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<tr>
<td>Average</td>
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<td>3.30</td>
<td>0.08</td>
<td>0.11</td>
<td>5.92</td>
<td>53.0</td>
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<td>0.490</td>
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<tr>
<td>Sample A</td>
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<td>3.38</td>
<td>0.07</td>
<td>0.11</td>
<td>6.35</td>
<td>47.2</td>
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<td>821.9</td>
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<tr>
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<td>4.29</td>
<td>0.08</td>
<td>0.39</td>
<td>6.64</td>
<td>46.6</td>
<td>90.3</td>
<td>0.506</td>
<td>904.6</td>
<td>830.8</td>
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<tr>
<td>Average</td>
<td>6.53</td>
<td>3.93</td>
<td>0.08</td>
<td>0.29</td>
<td>6.50</td>
<td>46.8</td>
<td>89.6</td>
<td>0.483</td>
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Table 5.8 Different case studies of bivariable functional dependency
Figure 5.1 3D Scatter plots of yield strength versus composition in the database
Figure 5.2 Representative micrographs showing the variation in size and volume fraction of equiaxed alpha and width of the $\alpha$ lamellae.
Figure 5.3 Scanning electron micrographs of the samples that have identical YS but different microstructural features
Figure 5.4 NN model predictions of YS (a) Microstructural features used as inputs, (b) Composition and microstructural features used as inputs
Figure 5.5 NN model predictions of UTS (a) Microstructural features used as inputs (b) Microstructure and composition used as inputs
Figure 5.6 Scatter plots of YS based on (a) Lath thickness and (b) O

Figure 5.7 Trend plots of YS on composition (a) Al (b) V (c) O and (d) Fe
Figure 5.8 Trend plots of YS based on the following microstructural features (a) VF of equiaxed alpha (b) size of the equiaxed α (c) width of α lamellae and (d) VF of total alpha
Figure 5.9 Trend plots of (a) YS and (b) UTS based on varying V and Al for different impurity contents
Figure 5.10 (a) 3D Scatter plot of YS based on wt% Al and equiaxed α size (b) Trend plots of YS based on equiaxed α and wt% Al for different O concentrations
Figure 5.11 Trend plot of YS based on (a) Al and V concentration with different Fe concentrations, (b) Al and V for different O concentrations

Figure 5.12 Trend plots of YS based on wt% V for two Fe concentrations (low Al, high O)
Figure 5.13 BSE images of samples, which are high in O and low in Al for different V contents (a-b) low V, and (c-d) high V content
Figure 5.14 Trend plot of YS based on wt% V for different Fe concentrations (low Al, and low O)

Figure 5.15 BSE images of (a-b) low V content and (c-d) high V content
Figure 5.16 Trend plot of YS based on wt% for two Fe concentrations (low Al and high O).

Figure 5.17 BSE images showing, (a-b) High fraction of secondary alpha and (c-d) Basketweave α in transformed β.
Figure 5.18 Errors associated with the bi-variable model

Figure 5.19 Composition and microstructure that gives same YS -Iso YS-surface
Chapter 6 Neural Network Modeling of Fracture Toughness of $\alpha+\beta$ Processed Titanium Alloys

6.1 Abstract

A rigorous integrated and recursive application of characterization, database population, and neural network modeling has resulted in the development of a fundamental understanding of the roles that microstructure, composition and continuum variables have on the fracture toughness of $\alpha+\beta$ processed Ti-6Al-4V. Several models have been developed to explore the influence of the individual microstructural features – a task that is often considered rather challenging given the complicated interplay between sample geometry, initial flaw size, material continuum response ($K$), and governing material parameters and micromechanisms. These models are based upon inputs that incorporate both continuum and micromechanistic details of the material. The former include both the local tensile properties (yield strength, reduction in area) and the local stress state at the crack tip through an inclusion of a plane-strain thickness factor while the latter are based upon quantified microstructural features and alloy composition, specifically the levels of Al, V, O, and Fe. The results differentiate which microstructural features (e.g., $\alpha$-lath thickness) contribute to modifications of the continuum variables which influence fracture toughness such as yield strength, and those which may influence both the continuum and the micromechanistic details of fracture (e.g., size of the
equiaxed α particles). Additional in-depth characterization of the material, including SEM EBSD, FIB site-specific 3D characterization and TEM analysis support the observations made using the models. Since the models are based upon an inclusion of an estimation of the plane-strain thickness, they capture not only the in-plane component of the stress state of the crack tip (\(\sigma_z\)) but also have as outputs both \(K_{IC}\) and \(K_Q\) values. This makes it possible to begin to estimate the effect of sample thickness on \(K_Q\) and explore the magnitude of the difference between a measured \(K_Q\) and \(K_{IC}\) values. Such results have significant implications for future understandings of the effect of microstructure on toughness and the ultimate prediction of toughness properties.

6.2 Introduction

The global material response to the presence of the crack (i.e., the fracture toughness, \(K\)) is an extremely difficult property to predict for a specific combination of microstructure and composition due to complicated interplay between sample geometry, initial crack size, the stress state at the crack tip and the response of the microstructure to the local stress state. Indeed, while there is a reasonable understanding of the relationships between applied stress, basic sample geometries and initial crack shapes/sizes and the resulting fracture toughness, there is a dearth of legacy knowledge regarding the influence of the individual continuum material parameters (e.g., yield strength and stress state at the crack tip, \(\sigma_z\)) and the response of the microstructure to the local stress state, including crack tip opening and growth on the measured toughness. This knowledge asymmetry is shown schematically in Figure 6.1 with the well-established connection between geometry and crack size and the material property shown
with a solid line. In a similar fashion, the various interconnected material variables are shown with dotted lines to indicate the lack of understanding of their interrelationships and contribution to the material response, $K$. This problem is sufficiently complex so as to have been largely ignored. Consequently, past research has typically focused on the inverse relationship often observed between the yield strength and the fracture toughness (see Figure 6.2). The data in this particular schematic has been compiled from a variety of sources and for a range of Ti-based alloys. Similar plots have guided alloy selection and fracture toughness knowledge for the past 35 years and are shown throughout the literature [78]. However, owing to the difficult nature of the interpretation of the material response and limited funding, there have been few efforts to move beyond this rudimentary observation to establish a mechanistic understanding of the influence of microstructure on the fracture of ductile multi-phase materials in general and for Ti-based alloys specifically.

An additional complication for both the design of components and the development of a micromechanistic understanding of fracture is associated with the variability of the local stress state at the crack tip. While Figure 6.1 appropriately labels the material response as $K$, the $K$-type that is determined is a function of the stress state at the crack tip, which in turn is a function of both sample geometry and local yield strength. It is well-known in the fracture toughness community that the stress state at the crack tip, specifically the in-plane stress parallel to the crack front – $\sigma_z$, can result in different fracture toughness values based on variations in sample thickness. This variability is clearly shown in Figure 6.3; the $K$ increases with decreasing sample
thickness, due to the domination of a plane stress condition within the sample. As can be seen, the radius of the plastic zone size ($r_y$) also plays a role. This size is related directly to the square of the ratio between the fracture toughness and the yield strength of the material in which the plastic zone resides, which can range from several microns to several millimeters, depending upon both $\sigma_{ys}$ and $K_{IC}$. From the Figure 6.3, one can see that the $K_{IC}$ value is the fracture toughness value of a material that is sufficiently thick such that plane strain dominates the test. However, it is often challenging to ensure that all tested samples result in a $K_{IC}$ value, rather than an apparent toughness, or $K_Q$ value. This is due to the fact that the threshold between plane strain and plane stress, and therefore between $K_Q$ and $K_{IC}$, is largely dependent upon the composition and the microstructure because they both influence tensile properties and toughness (i.e., $r_y \propto (K_{IC}/\sigma_{ys})^2$). Further complicating the problem for Ti-based alloys is the difficulty of ensuring that the heat treatment results in an average microstructure that is uniform over a length scale sufficient to measure a valid $K_{IC}$. For example, in this work, each coupon is thicker than 25 mm, yet not all have resulted in a valid $K_{IC}$. Thus, it is not difficult to imagine that the dimensions sufficient to achieve a valid $K_{IC}$ may be too great to result in a uniform microstructure for Ti alloys subjected to certain heat treatments.

These numerous challenges have severely limited the efforts to conduct studies, which detail the role of microstructure on toughness in ductile materials. The available data describing the role of microstructural features and composition on toughness in Ti-6-Al-4V provides a qualitative description (e.g., +/-0) of variables which influence fracture toughness, but such work does not directly distinguish whether such variables influence
toughness by influencing the continuum (e.g., yield strength) or by influencing the micromechanisms (e.g., crack path) [21, 79, 80]. Additionally, the work of Ritchie and Thompson has shown not only the importance of considering both macroscopic (continuum) and microscopic contributions to the fracture toughness in some materials (not Ti-based), but also that the two are interrelated and must be considered together when trying to develop an understanding of crack initiation and crack growth in ductile materials [81]. Several researchers have focused on materials where there are clear microstructural features responsible for crack/void nucleation such as hard intermetallics along grain boundaries. While other authors have followed an approach similar to that of Ritchie and Thompson, most have strictly worked to apply continuum theories to microstructural features [82, 83]. Such efforts have, by necessity, relied on unverifiable assumptions regarding the properties of individual phases, without considering the complex interrelationship between the phases, or even of variations in interfacial energy or the constraint provided by the local phases resulting in strengthening of the phases. An advantage that is afforded by an approach using a combination of neural network models and in-depth characterization is the simultaneous consideration of both continuum and microscopic contributions, allowing an analysis of their interrelationships (e.g., a microstructural features contribution to the strength which is often associated with the continuum approach), as well as their distinct differences (e.g., the contribution of a microstructural feature to microcracking or void nucleation).

Fortunately, there have been recent efforts to investigate such interrelationships using neural network approaches. The separation of the continuum and micromechanistic
contributions of the material to the fracture toughness may be possible using such neural networks. Authors have developed such a modeling approach for the prediction of tensile properties in both $\beta$ and $\alpha+\beta$ processed Ti-6Al-4V [10, 76, 80]. These approaches have been based on neural network models developed by McKay that incorporate Bayesian statistics [63, 64] and include accurate descriptions of the microstructural features based on rigorously developed stereological methods, as described elsewhere [59]. Following training and testing, the models for the prediction of tensile properties have resulted in differences of less than 2.5% magnitude from experimentally measured values for both yield strength and ultimate tensile strength [59]. Considering the extended range of tensile properties possible in Ti-6Al-4V (e.g., 725-930MPa for yield strength), a prediction with an associated uncertainty of ±2.5% is quite remarkable.

In addition to their ability to make blind predictions, such models may be used to perform virtual experiments, where a single input parameter, such as a microstructural feature or solute content, is changed while all other inputs are kept fixed at some value (e.g., their average). These virtual experiments, so called because they might otherwise be impossible to achieve in the laboratory, given that the microstructural features are often very complex and interrelated, can be used to probe the functional dependencies of certain microstructural features on the mechanical properties. While such results have been quite successful in the prediction of tensile properties, the application of neural network models to the prediction of fracture toughness is expected to be more difficult, and hence, the results potentially less accurate. This is due to the complicated manner in which the material responds to the application of a load in the presence of a flaw. Indeed,
the material response and the absence of a sufficient legacy data on the influence of microstructure on fracture will make verification of results a challenge. Therefore, following the virtual experiments, additional characterization focused on further developing an understanding of the fracture mechanisms related to microstructure and composition has been performed.

The model also include a factor that can be used to estimate the thickness of the plane-strain region of the specimen, and to predict a first approximation the expected plane strain thickness without necessarily knowing the yield strength of the material. The direct inclusion of the plane strain region is important for two reasons. First, it allows a direct analysis of $K_Q$ as a function of thickness, and the comparison of $K_Q(t)$ with valid $K_{IC}$ values. This is due to the fact that the rules-based model not only “scales” the $K_Q$’s to reflect values closer to their $K_{IC}$ if plane-strain, rather than mixed-mode, conditions dominated the test, but also “adjusts” $K_{IC}$’s to be $K_Q$’s if the plane strain thickness were to be decreased. Second, it adds a physical constraint as an input variable to the virtual experiments, thereby more closely resembling the actual experiment. The plane strain thickness is proportional to the yield strength of the samples in the database. The direct inclusion of this thickness also reduces the bias given to the yield strength by neural network model. An additional practical and important consequence in the prediction of an appropriate thickness that would result in a test dominated by plane strain, and hence $K_{IC}$, is the possibility to predict, and hence design to, a $K_Q(t)$ where the stress state at the crack tip is positively (and predictably) influenced by the triaxality at the crack surface, rather
than an overly conservative $K_{IC}$ for thin specimens, resulting in smaller (thinner) components and a total weight savings.

Therefore, this chapter has three principal aims. The first is the development of the necessary databases to relate composition/microstructure and fracture toughness in $\alpha+\beta$ processed Ti-6Al-4V. The second is the development of rules-based models to predict the fracture toughness in these alloys. The last is the determination, validation and interpretation of the functional dependencies of the models. It is necessary to develop several types of models to accomplish these tasks and to begin to develop a mechanism-based understanding of fracture in $\alpha+\beta$ processed Ti-6Al-4V. These are shown schematically in Figure 6.4 (a-c) where the inset double triangle captures the interconnected nature of the continuum material variables such as the yield stress and stress state at the crack tip, and their dependencies upon microstructure and composition, the micromechanistic variables that may be involved in fracture. Notably, the extrinsic toughening mechanisms are neglected as they can likely be related to individual microstructural features and therefore contribute as identified micromechanisms. The models that will be developed include a baseline model which only includes the continuum variables thereby approximating the legacy understanding (see Figure 6.4 (a)), a model which only includes potential micromechanistic details (see Figure 6.4(b)), and a model which includes both the continuum and micromechanistic details, including microstructure and composition (see Figure 6.4 (c)). These models, their quality, and their functional dependencies will be shown and the results discussed.
6.3 Experimental Procedures

In keeping with the previous work on the prediction of tensile properties, this research includes an intentional variation of not only microstructural features but also composition. A total of nine different Ti alloys (based around Ti-6Al-4V) were produced with intentional variations in the relative amounts of the individual elemental species, including the impurity Fe and O contents. The variations in alloy compositions, as measured by TIMET North American Technical Laboratory, Henderson, NV using ICP, are: Ti-xAl-yV (4.76<x<6.55; 3.30<y<4.45) with controlled variations in the impurities O and Fe (0.07<wt% O<0.20; 0.11<wt% Fe<0.41). For reference, the AMS specification #4920 is: 5.5<wt% Al<6.75; 3.5<wt% V<4.5; wt% O(max) = 0.20; and wt% Fe(max) = 0.30. The Al content ranged below that of standard Ti-6Al-4V, while the V exhibited a slightly extended range. The maximum Al content was intentionally kept below the maximum allowed by the standards in order to avoid the onset of short-range ordering of the HCP α phase that could result in embrittling α₂ domains. The O level had a slightly extended range, notably with an alloy that was O lean (0.07 wt% - 700 ppm), even with respect to ELI grades (0.13 wt% max). The Fe level also had an extended range. For each of the nine alloy compositions, six samples were exposed to different thermomechanical processing histories, producing a total of 54 fracture toughness samples. It should be noted that in the preparation of these samples, all were taken from the same radius of a round billet, minimizing the differences in strain during deformation and also any potential differences in texture. For each fracture toughness sample, there
was a corresponding tensile sample, as documented in the previous work, with nominally the same microstructure.

The geometry of each fracture toughness specimen was that of a compact tension specimen with a fatigue precrack. These specimens were tested at room temperature according to ASTM E399 specifications. The thickness ($B$) was 25.4 mm, and the $W$ and $a$ values were ~ 51 mm and 20.5-25 mm, respectively. These geometries were selected based on an estimation that the tests would yield values that would qualify as $K_{IC}$. However, only 23 tests resulted in valid $K_{IC}$ measurements. The other tests have been reported as $K_Q$. It should be noted that of the 24 samples with the highest O contents, all but one were reported as $K_{IC}$, and consistently had lower fracture toughness values. This is not surprising given the fact that these samples also invariably have higher yield strengths and lower ductility. However, given the variation in type of toughness reported, the models developed in this paper will be for the experimentally less rigorous $K_Q$. It is expected that the normalization of the models with the inclusion of the plane strain thickness might give a $K_Q$ that is a reasonable approximation of $K_{IC}$.

Following fracture toughness testing, an undeformed section of the grip from each sample was excised for metallographic preparation. Following traditional metallographic sample preparation techniques, the samples were characterized using a FEI/Philips Sirion scanning electron microscope (SEM) operating in backscattered mode at 15 kV with a resolution of approximately 3.0 nm. Each image had an area of 3872 x 2904 pixels with a depth of 8 bit (pixel resolution ~ 25nm/pixel). The high resolution afforded using the SEM, especially when compared with other available techniques (e.g., optical
micrographs), is of paramount importance because it is essential to provide the highest possible fidelity quantified microstructural data to the neural networks. The specimens were imaged at four random locations to avoid overlap and edge effects. Microstructural features present in the four micrographs were quantified using the stereological techniques [59]. These features include the equiaxed alpha size (equiaxed-$\alpha$ size, $\mu$m), the volume fraction of equiaxed $\alpha$ ($F_{V^{equiaxed-\alpha}}$), the volume fraction of total alpha ($F_{V^{total-\alpha}}$), and the width of the $\alpha$ laths in the transformed $\beta$ regions using Gundersen’s approximation ($\alpha$-lath width, $\mu$m) [59, 75]. These microstructural features, and the corresponding compositions, were first used as inputs to predict the yield strengths of the samples, based on the model developed in previous work [80]. The results were then compared to tensile specimens that had nominally the same thermomechanical processing history, and therefore, nominally, the same microstructure and properties. Figure 6.5 shows the predicted yield strengths based on the microstructure and composition present in the fracture toughness specimens along with the experimentally measured yield strengths from a corresponding tensile test. As can be seen, all of the blind predictions resulted in data that falls within ± 5% of the data obtained from the corresponding tensile tests, further validating the previous work [80]. Thus, while either the experimentally determined or the predicted yield strengths can be confidently used as inputs in the models, the experimentally measured yield strengths and experimentally measured reduction in area are used for the majority of the models to reduce the possibility of compounding uncertainties between models.
A database containing experimentally determined data was developed. It includes: alloy composition, microstructure, tensile properties (yield strength and reduction in area), a factor that scales with plane strain thickness, and fracture toughness for subsequent analysis. The plane strain thickness factor is calculated from an average of 5 measurements across the fracture surface, as illustrated in Figure 6.6. This measured plane strain thickness is proportional to the experimental yield strength of the specimen. The database was analyzed using an artificial neural network with the Bayesian architecture, after the work of D. Mackay [61, 64]. This probabilistic neural network was used to develop 12 different models to predict the fracture toughness of the alloy Ti-6Al-4V based on varying combinations of microstructure, composition, yield strength, reduction in area, and plane strain thickness. Of these 12 models, two sets of models have been developed – four models that only include the combinations of the three continuum contributions mentioned above (YS, RA, \( t_{\text{plane strain}} \)), and eight models that also include microstructure and composition. In this way, it is possible to explore separately the influence of microstructure/composition on fracture toughness through its modification of continuum variables (e.g., yield strength) and of microstructure/composition on fracture toughness through extrinsic means. In addition, two models were developed to predict the plane strain thickness. One of these two types directly included composition and microstructure, while the other included yield strength and reduction in area.

Typically, when the neural network is being trained and tested, one particular model emerges as a superior model, based on the minimization of the residual error using a mean square error (MSE) approach with respect to the experimental values. When this
superior model is further interrogated for the determination of the functional dependencies, each of the dependencies will represent a physical reality that is easily interpreted. For example, the influence of O on the yield strength in α+β processed Ti-6Al-4V emerges as the most potent strengthening mechanism, which fits with the legacy data. However, as described previously, the absence of legacy information relating microstructural features with fracture toughness properties, in addition to the complex nature of fracture in general, creates a situation where it is appropriate to consider more than one model. It is often useful to compare several models because each model gives different significance to different variables. Therefore, for each of the 14 types of models (12 predicting fracture toughness, two predicting plane strain thickness), a total of 288 models were developed with different seed values, number of nodes, and sigma widths. Of these 288 models, at least best three models with the lowest MSE values were subsequently investigated.

The development of these models has been described elsewhere. Once developed, the neural network can be used in one of two ways. The first involves the development of a predictive tool, where the properties of a test dataset are predicted. The test dataset represents a fraction of the 54 samples that were not used for the training, in this case, 38 samples were used to train the model and 16 to test the model. The second involves the use of the neural network to perform virtual experiments. These are usually control experiments where the values of given microstructural features can be set to, or held at, given values. In this way, it is possible to obtain information from virtual experiments where such control of individual microstructural features may not be possible.
experimentally. One such virtual experiment is the determination of the functional dependencies. The functional dependencies have been determined by setting all but one variable at a fixed average value, and allowing the variable to vary while observing the change in the predicted fracture toughness. The predicted fracture toughness can then be plotted directly against this single variable, and can provide insight into the functional dependency of the variable.

As will be observed, the functional dependencies as determined from the virtual experiments are sometimes quite complex. Therefore, to begin to validate selected dependencies, specific samples were sectioned to characterize the microstructure associated with the fracture surface. From each selected sample a region from the centerline (plane strain) was sectioned using wire electrical discharge machining (EDM). These samples were prepared using conventional metallographic techniques, and polished through 0.05 µm colloidal silica. They were imaged using the FEI Sirion SEM operating in backscattered mode at 15 kV with a resolution of approximately 3.0 nm. SEM mosaics including the last portion of the fatigue pre-crack as well as the first 2 mm of fracture were prepared from the samples. A stereological procedure was developed to estimate the fraction of the fracture surface associated with the equiaxed alpha. This procedure measures the ratio of the length ($S_V^{\text{equaxed-\alpha}}$) of fracture surface associated with equiaxed alpha to the total fracture interface ($S_V^{\text{total}}$). Selected locations of particular interest, including those exhibiting microstructural details associated with the fracture surface (e.g., microcracking, crack-induced voids, deformation structures in transformed $\beta$), were further analyzed using electron back-scattered diffraction (EBSD) techniques in
a Philips XL-30 ESEM with TSL software. Site-specific TEM foils were prepared at regions of interest such as microcracks using FEI’s Nova 600 small dual-beam focused ion beam (DB-FIB) microscope. TEM analysis was conducted using various TEM’s, such as the FEI CM 200, and the FEI Tecnai TF20 microscope operating at 200 kV and a probe-corrected FEI Titan3 80-300 microscope operating at 300 kV. Additionally, site-specific features were extracted using the FEI Nova 600, and then analyzed using Slice-and-View™ software to explore the three-dimensional nature of certain features of interest.

6.4 Results and Discussions

The three backscattered scanning electron micrographs shown in Figure 6.7 (a-c) clearly illustrate the difficulty often faced by researchers when attempting to isolate the interdependent effects of alloy composition, microstructure, and their combined effect upon tensile properties, crack-tip opening displacement/stress state, and toughness. These three micrographs correspond to three different samples of the alloy Ti-6Al-4V, with intentional variations in alloy composition and thermomechanical processing, resulting in statistically different microstructural feature sizes and spatial distributions (see Table 6.1). However, these three samples have nominally identical toughness properties \(K_Q \sim 83 \text{ MPa}\sqrt{\text{m}}\). Notably, the microstructures shown in Figure 6.7 (a-c) have resulted in samples whose yield strengths vary by more than 12% (725 MPa to 832 MPa). This variation in yield strength for the same fracture toughness is particularly interesting as it is nominally equivalent to the 12% scatter bands for plots of stress intensity for failure as a function of tensile yield strength for annealed Ti-6Al-4V [84]. This indicates that the
unknown microstructures and their variability have a significant influence upon the scatter observed in the observed toughness. The implications of this figure coupled with the yield strength variability in the legacy data clearly indicate that while the yield strength may exhibit a strong influence on the fracture toughness, the microstructure affects the fracture toughness in additional ways. Therefore, in addition to modifying the local tensile properties and hence the stress state at the crack tip through solid solution strengthening and slip length modifications, microstructure must also affect extrinsic phenomena or micromechanisms such as the crack path modification, all shown schematically in Figure 6.1. Thus, one can easily understand the difficulty in separating the continuum and micromechanistic details of fracture based upon this specific example in which, the total effect on properties resulting from the collective and offsetting changes in composition, size and distribution of microstructural features, and local tensile properties (themselves determined by microstructure and composition) is negligible.

The results which follow show how the effects of continuum contributions and microscopic contributions may be isolated by comparing the functional dependencies obtained using different model architectures. While the continuum variables that influence toughness include yield strength, sample geometry, crack size, and the resulting crack tip fields, for the purposes of this analysis the continuum inputs are limited to yield strength, ductility, and a factor that scales with plane strain thickness. Indirectly, these continuum variables will allow for the investigation of the effect of crack tip fields (e.g., plane strain vs. mixed mode vs. plane stress conditions) on the reported value of $K$ (i.e., $K_{IC}$ vs. $K_Q$).
6.4.1 Part 1: Development and Interpretation of Models for the Prediction of Fracture Toughness

Several models were developed with various inputs; a few of the best models chosen based on their test errors. Predictions were made to examine how well the experimental values aligned with the predicted values. The quality of the models was decided based on the analysis of the mean square of the errors (MSEs). This analysis often helps to separate seemingly similar models, and is often used to pick “the optimum” model. The average and maximum deviations ($\delta$) and errors (E) are reported from model predictions. The three models with the lowest MSE for each of the 12 model types have been averaged and are reported.

Figure 6.8 (a-d) show the predicted vs. experimental toughness for four representative models while Table 6.2 summarizes the numerical analysis of all 12 models types, with the four shown in Figure 6.8 highlighted for ease of comparison. Figure 6.8 (a) shows the best model that excludes a direct input of microstructure and composition, specifically the model with three continuum variables included. This model has a maximum deviation from experimental of 19.5% and an average deviation from experimental of 3.5%. Although this is the best “baseline” model, which represents the legacy understanding of toughness in Ti-based alloys (i.e., it only includes continuum effects and does not directly include either microstructure or composition), it is a considerably poorer model than those that include microstructure and composition inputs. Consider Figure 6.8 (b-d), which either includes microstructure and composition without any direct inclusion of continuum inputs (see Figure 6.8 (b)) or includes continuum
inputs in addition to composition and microstructure, such as yield strength \((Figure 6.8 (c))\) or yield strength, plane strain thickness, and reduction in area \((Figure 6.8(d))\). These three models, in addition to having superior MSEs (15.6, 10.2 and 10.6 respectively), have significantly lower average and maximum deviations than predicted \((\delta_{\text{avg}}: 1.8, 1.1, 0.9; \delta_{\text{max}}: 7.9, 5.6, 5.8)\), compared to the model shown in Figure 6.8 (a).

The results of these numerical analyses clearly demonstrate two significant factors when considering the development of tools for the prediction of the toughness of a material. First, in addition to the inclusion of continuum variables it is imperative to include details regarding microstructural and compositional inputs as these reduce uncertainty and improve the quality of the models. Indeed, excluding microstructure and composition (i.e., focusing only on the influence of yield strength) will lead to significant scatter in the predictions of fracture toughness. These models indicate that such scatter is rather significant; the raw data obtained for this research indicates that such scatter exceeds 20%. Second, of the models that do include compositional and microstructural inputs, the models that also directly include yield strength are far superior to those that do not. When considering a further analysis of the mean square errors (which average from 10.2 to 15.6), it is apparent that the mean square of the errors is less than 11.52 for all the models where yield strength is included; those that did not directly include yield strength had MSE’s greater than 12.89. Similarly, an analysis of the maximum deviation \(\delta_{\text{max}}\) shows that the four models that include yield strength in addition to the composition/microstructure have an average maximum deviation of 6.25%, while the four models that exclude yield strength but include composition/microstructure have an
average maximum deviation of 8.65%, indicating a poorer quality model. These observations reinforce the importance of considering the continuum effect of yield strength on establishing the stress state at the crack tip, independent of the effect, if any, that specific local microstructural features have on the micromechanisms of toughness. However, it is equally clear that the inclusion of microstructure and composition as inputs leads to improvements in the models; the models may then be interrogated to help determine possible mechanisms.

It is important to explore the effects that the individual input variables have on toughness. However, as previously described, this is particularly challenging as the fracture phenomenon is a complicated interplay of many variables that control various aspects of fracture mechanisms such as crack propagation or possible extrinsic toughening/weakening mechanisms. Therefore, rather than only focusing on the best models (i.e., Model #6 or #12), a series of functional dependencies have been developed for each of these models in order to compare them and thus explore the effect of the microstructural and compositional variables on toughness – both in conjunction with and isolated from the continuum variables. As will be seen, this novel simultaneous application of different neural network architectures to such a complex problem has resulted in some newly developed understandings of the contributions of microstructure to fracture toughness in α+β processed Ti-based alloys.
6.4.2 Modeling the Influence of Continuum/Macroscopic Variables on Fracture Toughness

Effect of Yield Strength on Toughness: The functional dependencies show a significant decrease in $K_Q$ with increasing yield strength for all models that include yield strength as input variable as well as compositional and microstructural variables. Figure 6.9 (a-c) shows three such models. This decrease in $K_Q$ is consistent with the legacy data, and therefore, is not a surprising observation. This relatively simple decrease is analogous to the trend observed in previous work on the effect of composition and microstructure on the tensile properties of $\alpha+\beta$ processed Ti-6Al-4V. Additionally, this figure includes trends calculated from the three best models. These trends clearly show that the functional dependencies obtained for each model are quite similar, indicating that the models have learned from databases, identified a strong dependency and established a nearly identical weighing function. This would be expected to result from the data with strong correlations. Thus, another advantage of the model comparison framework emerges; as an assessment tool for the accuracy of individual functional dependencies. As will be shown below, the functional dependencies for the “best” models often exhibit such remarkably consistent trends, clearly indicating the importance and influence of a particular input variable. However, it is also observed that in some cases the forms of the functional dependencies are rather different among the “best” models. Such differences were often observed in conjunction with either an inconsequential dependency (i.e., minimal influence of a feature upon a property), or an unusually complex dependency,
including several that appear to exhibit second or higher order natures, indicating a likely over-fitting of the models to a particular variable.

It should be noted that Figure 6.9 (c) includes an estimation of plane strain thickness as input. When compared with Figure 6.9(a-b), the slope shows a significant decrease (~40%) in the degree to which increasing yield strength decreases toughness. The experimental yield strength is proportional to measured plane strain thickness. This will indicate the effect of yield strength on the local stress state. Recall that yield strength influences the stress state (\(\sigma_z\)) at the crack tip, and hence, whether \(K_{IC}\), a lower value, or \(K_Q\), a higher value, is experimentally determined for a given thickness. When the plane strain thickness is included directly (Figure 6.9(c)), the corresponding component of the influence of yield strength on toughness would diminish. This is because the plane strain thickness shows a functional relationship with yield strength. When plane strain thickness is added as an input, even though it has a functional relationship with the yield strength, the model assumes a less bias to yield strength. This causes a decrease in magnitude of the effect of YS on \(K_Q\) and can be confirmed in Figure 6.9 (c). It is also important to note that the functional dependency of YS on \(K_Q\) when plane strain thickness as included as input is reasonably with in the limits of experimental data.

Interestingly, as can be clearly seen, yield strength still has a significant effect on toughness independent of the effect of the stress state at the crack tip. Including reduction in area in the model does not significantly decrease the effect of yield strength on toughness in the absence of plane strain thickness; therefore the contribution of yield strength might not be solely attributable to a stress-strain argument. Yield strength must
affect the toughness in additional ways. The exact source of this trend (decreasing toughness resulting from increasing yield strength) is not known for this two-phase, elastic-plastic, strain hardening material. However, the authors suggest that one of the following phenomena occurs. First, it is likely that the crack tip opening displacement is smaller for samples with higher yield strengths. It can be shown that the stress distribution remains higher for a greater distance in front of the crack tip as the radius of the crack tip decreases. This could result in a greater volume of material experiencing a higher stress, which, if above a critical stress (e.g., a critical resolved shear stress for dislocation motion), then it would be possible to accumulate damage in front of the crack tip (e.g., pore or crack nucleation), resulting in a lower toughness. It is also possible that there exists either a microstructural or compositional variable that decreases the damage tolerance of the material immediately ahead of the crack tip (e.g., a reduction of the critical stress to cause pore or crack nucleation) that is inversely proportional to yield strength, yet does not significantly influence either yield strength or ductility in a negative fashion. Lastly, if ligament bridging were occurring, the yield strength of the material would be directly proportional to the crack tip radius and to crack propagation. It is also quite possible that all these phenomena are occurring. However, the determination of which of these operative mechanisms are operative is not trivial, and has not been determined in this work.

*Effect of Plane Strain Thickness on Toughness:* While the plane strain thickness does modify $K_Q$, the magnitude of this contribution is often not clear. A classical estimation of $\beta_{1C}$, shown by Irwin as an empirical relation based upon a plastic zone size calculation
(and hence, upon a rigorous determination of both $K_{IC}$ and yield strength), would result in a $K_Q$ which does not approximate, indeed, far underestimates the $K_Q$’s obtained in this work for this particular alloy. Thus, it is still a challenge to determine the influence of thickness upon the measured toughness ($K$) and whether that toughness represents a valid $K_{IC}$ or plane stress dominated $K_Q$. It is equally challenging to determine the appropriate experimental conditions based upon classical fracture mechanics, as the approximation for $B$ ($B \geq 2.5(K_{IC}/\sigma_{ys})^2$), represents a minimum thickness before plane strain behavior occurs, and not an absolute prediction that it will occur. The variation in whether plane strain behavior occurs must therefore be related to intrinsic material variability, including the effects of both microstructure and composition. Consequently, by including both $K_Q$’s and $K_{IC}$’s in this model, as well as a factor to incorporate the variation in plane strain thickness, the researchers have shown it is possible to estimate the impact of thickness on the variability on toughness that exists between $K_{IC}$ and $K_Q$.

Consider Figure 6.10, which presents the normalized influence of plane strain thickness on $K_Q$ when yield strength is included in the models, and is held at a fixed “average” value. In this figure, the samples with a smaller plane strain thickness (i.e., effectively those with $K_Q$ values) exhibit predicted toughness values up to $\sim 9$ MPa√m greater than their $K_{IC}$ microstructural/compositional analogs. As was done with the effect of yield strength, the functional dependencies and their predicted errors obtained using the three best models are shown to illustrate the similarity among them. Indeed, the results show a negligible difference between the three best combinations seed values, nodes, and sigma widths indicating a very clear effect of variable on property. The
influence of plane strain thickness on the toughness suggests that the magnitude of the difference between a true $K_{IC}$ and a measured $K_Q$ can be up to at least 10% of the value for $\alpha+\beta$ processed Ti-6Al-4V. The interplay between yield strength, plastic zone size, sample thickness, and $K_Q/K_{IC}$ still represents a complex problem – but this work allows for a first approximation of their interdependencies.

**Modeling the influence of Microstructure and Composition on Fracture Toughness:**

The use of artificial neural networks and virtual experiments allows for exploration of the mechanisms by which microstructural features affect fracture toughness (e.g., through modifications of properties that must be considered from a continuum perspective or through modifications of microscopic contributions such as crack path or “extrinsic” toughening mechanisms). Three of the microstructural features that affect fracture toughness are the volume fraction of equiaxed alpha, the size of the equiaxed alpha, and the thickness of the $\alpha$ laths in the transformed $\beta$ regions. For a model that includes only composition and microstructure, the three functional dependencies are shown in Figure 6.11 (a-c). Clearly, the volume fraction of equiaxed alpha significantly affects the toughness ($\sim 20 \text{ MPa}\sqrt{\text{m}}$), while the size of the equiaxed alpha has a lesser effect ($\sim 7 \text{ MPa}\sqrt{\text{m}}$), followed by a negligible effect of the thickness of the alpha laths ($\sim 1 \text{ MPa}\sqrt{\text{m}}$). However, when the yield strength is included as a separate input variable, the predicted functional dependencies (see Figure 6.12 (a-c)) for important microstructural features on toughness differed in predictions. Consider the dramatic reduction in the degree to which toughness is dependent upon the volume fraction equiaxed alpha when yield strength is included as a variable in the model. In a model that includes yield
strength, the contribution of volume fraction of equiaxed alpha becomes negligible (~ 0 to 1 MPa√m). This strongly suggests that the volume fraction of equiaxed alpha only affects toughness by modifying the macroscopic yield strength of the material. The effect of the volume fraction of equiaxed alpha on the tensile properties is considered in the last chapter and it is the important variable that affects tensile properties. Similarly, consider the negligible reduction in the degree to which toughness is dependent upon the size of the equiaxed alpha when yield strength is included. In the model that includes yield strength, the contribution of the size of the equiaxed alpha has the greatest effect (~7 MPa√m). This suggests that the equiaxed alpha size (or another microstructural feature directly linked to the size of the equiaxed alpha) has, at the very least, as significant an influence on the micromechanisms of fracture as it does on the continuum variables. The manner in which this feature affects toughness is further discussed in a subsequent section. Lastly, consider the influence of effect of the thickness of the alpha laths, in models with and without yield strength. While it does change from a negative to a positive slope (~1 MPa√m), such a minimal influence suggests that it does not modify the micromechanisms significantly, and only slightly influences toughness. The inclusion of yield strength in the model reduces the bias given to the composition and microstructural features and also gives insight into which features contribute to the continuum and which features are directly related with micromechanisms.

In a similar fashion, the functional dependencies of the compositional variables have been determined, and their influence upon the fracture toughness has been explored. The functional dependencies obtained for a model including microstructure and
composition, but not yield strength, are shown in Figure 6.13 (a-d); the functional dependencies obtained for a model including microstructure, composition, and yield strength are shown in Figure 6.14 (a-d). Two primary observations can be made from these figures. The first observation is the manner in which both \( \alpha \)-stabilizers (Al and O) influence the toughness. These functional dependencies exhibit relatively clear trends for the model without yield strength as an input (Figure 6.13 (a,b)). The trends are fairly consistent for several of the best models. This is expected, given the strong positive influence of Al and O on yield strength, as well as the strong negative influence of YS on toughness. However, the implications of the functional dependencies are less clear for the model that includes yield strength. The trend plot of toughness versus Al content looks different when YS is included in the inputs. Additionally, the uncertainty in the model predictions, which is characterized by error bars, is significantly different. The error bars become large at low and high O compositional space. The decrease in magnitude of the effect of O on toughness when yield strength is added as an input suggests a functional relationship between O and yield strength. It should be noted that the raw data, which has already produced exceptionally clear dependencies of the composition on the tensile properties, is taken from within the same billets as these toughness specimens. The second observation is the manner in which both \( \beta \)-stabilizers (V and Fe) influence the toughness. The functional dependencies for each of these species show an increase in toughness with increasing solute content for models that exclude yield strength. Additionally, although V appears to have a neutral effect on yield strength, it has little positive influence on toughness. This trend is consistent for the models that both include
or exclude yield strength. Fe has a positive influence on toughness in all of the models. Lastly, the Fe trends are contrary to the previous observations; which clearly showed an inverse relationship between strength and toughness for the other seven compositional or microstructural variables. Fe is the only variable that causes an increase in both yield strength and toughness as its concentration increases. Therefore, this anomalous observation must be explored further.

6.4.3 Part 2: Characterization to Further Validate and Explore Functional Dependencies

Effect of Equiaxed α Particle Size:

As described above, the microstructural feature that appears to be associated with the most significant influence on fracture toughness ($\Delta K_Q \sim 5-10 \text{ MPa}\sqrt{\text{m}}$) via micromechanisms is the size of the equiaxed alpha grains – either directly or through a related microstructural feature. The effect of size of the equiaxed alpha on fracture toughness is inline with the literature observations. It is suggested that size of the equiaxed alpha is associated with the roughness of the crack front, which increases, with increase in size of the equiaxed alpha. When the size of the equiaxed alpha grain is higher, then the roughness of the crack front profile dominates the inherent crack propagation resistance term. This effect is clearly understood from the trend plots. The inherent resistance to crack propagation is included in the models in terms of continuum inputs. However, the trend plot of toughness on size of the equiaxed alpha has not changed indicating no effect coming from inherent crack propagation resistance.
To confirm the observed functional dependency and the implications discussed previously, selected samples (see mosaics in Figures. 6.15 (a-b) of high resolution SEM micrographs) were analyzed to determine whether cleavage of equiaxed alpha particles was present and to also measure the fraction of the fracture surface that was associated with equiaxed \( \alpha \) particles (\( \% S_{V\text{-equiaxed-} \alpha} \)). Stereological procedures were developed to measure the fraction of fracture surface occupied by the equiaxed alpha particles. The results clearly show an absence of measurable cleavage within individual equiaxed alpha particles. Additionally, the results of the surface fraction analysis have been compared directly with the bulk stereology measurements of equiaxed alpha size and fraction and are shown in Figures 6.16 (a-b), respectively. While the plot of equiaxed \( \alpha \) particle size (Figure 6.16 (a)) does not show a clearly discernable trend, the data is scattered. However it shows the decrease in fraction of fracture surface occupied by the equiaxed alpha with increase in particle size. The equiaxed \( \alpha \) fraction measured from the cross section of the fracture surface shows a clear trend with the bulk volume fraction of equiaxed alpha particles (Figure 6.16 (b)). This latter plot appears to be defined as a maximum surface area equivalent to the bulk volume fraction, with scatter in data suggest that the bulk volume fraction may be larger than the fraction of surface occupied by the equiaxed \( \alpha \) particles. This result is not surprising, and may be rationalized by considering that the fracture surface is half of a mating surface. Thus, one cannot readily determine whether the fracture surface followed an equiaxed \( \alpha \)/equiaxed \( \alpha \) boundary (a \( F_V^\text{point-count} = \frac{1}{2} \) on both, totaling 1) or an equiaxed \( \alpha \)/transformed \( \beta \) boundary (\( F_V^\text{point-count} = \frac{1}{2} \) on one, zero on the other, totaling \( \frac{1}{2} \)). In other words, an analysis of the bulk volume fraction equiaxed
$\alpha$ particles will be equal to or greater than the traction analysis. It is also important to note that there exists significant scatter in data for each of the plots. This scatter is due to the fact that the other variables are simultaneously changing – therefore, the surface area of non-intersecting spherical particles, calculated using the stereologically measured volume fraction and size is compared against $\%S_{V}^{\text{equiaxed-\alpha}}$ in Figure 6.17. While this simplification is known to not be a rigorous analysis, it may be used to approximate what may be occurring. This plot does show that as the surface area of the equiaxed $\alpha$ particles increases, the fraction of the fracture surface occupied by the equiaxed alpha particles also increases, which validates the functional dependency observed for equiaxed $\alpha$ particle size and supports an interfacial dominated fracture mechanism. However, it does not explain why there is not a greater functional dependency for the volume fraction of the equiaxed alpha particles. A consideration of the interplay between size, volume fraction, and the two types of surfaces (particle-particle and particle-transformed matrix) leads one to the conclusion that for these volume fractions (>35-40%), percolation theory is valid, and there should be a greater increase in surface area by modification of the equiaxed alpha particle size (i.e., an increase in particle-particle surface area) rather than a volume fraction change (i.e., a small change in particle-transformed matrix surface area).

Microcracking and equiaxed alpha particles

Fractured specimens were prepared in cross-section and characterized to further investigate the role of the equiaxed alpha particle size on fracture mechanisms and the observed toughness of the material. The backscattered scanning electron micrographs
shown in Figures 6.18 (a-c) are representative micrographs of subsurface microcracking that were observed frequently in many samples. The majority of the sizable cracks that were observed could be easily associated with the equiaxed alpha particles. While Figures 6.18 (a-b) show cracks in the early stage of formation or growth, Figure 6.18(c) shows the ductile nature of fracture for a large crack within the equiaxed alpha particles. Figure 6.19 shows microvoid formation and coalescence between two equiaxed particles in a particle cluster. This phenomenon is widespread in steels, which have carbide particles in it and microvoids originate at carbide particles. It is interesting to observe these voids at equiaxed alpha particle boundaries even though Ti alloys does not have carbides. It is important to probe these boundaries because apart from microvoids, the majority of the large cracks that exist are associated with an internal structure of the equiaxed alpha particles and are not associated with the boundary between the particle and the transformed β, nor are they often associated clearly with existing α/β boundaries.

The distinct contrast that is observed within the alpha particle clusters is typically associated with channeling contrast in backscattered electron micrographs, indicating the presence of an orientation difference and the possibility of a boundary delineating such an orientation difference. The presence of such boundaries has been confirmed using EBSD techniques. An EBSD orientation micrograph consisting of a combined alpha inverse pole figure and image quality map is shown in the backscattered SEM micrograph shown in Figure 6.20 (a). The image quality map is shown separately in Figure 6.20 (b). These micrographs capture the same region of interest as shown in Figure 6.18 (b), but rotated to 180° rotation. The dark regions in Figure 19 (a-b) are
associated with points where the quality of the EBSD signal is poor and hence the analysis exhibits low confidence. Often, these are associated with boundaries of some type, as in this case. It is clear from these figures that the crack has followed a path delineated by misorientation, indicating the presence of boundary. It should be explicitly noted that based on similar observations on such clusters in previous work, stereological methods that were used to determine the average size of the equiaxed alpha particles. So database population was based on the concept of particle clusters, rather than large discrete particles, and used channeling contrast to help determine particle size. Therefore, the particle size conducted for the database and subsequent analysis does include accurately these orientation boundaries.

Additional EBSD analysis shows a predominance of microcracks occurring along equiaxed alpha particle surfaces between neighboring particles, which has nearly parallel basal planes. The microcracks appear to follow types of twist boundaries about the \([0001]_\alpha\) axis. Four such examples are shown in Figures 6.21 (a-d), with the crystal overlaid on the EBSD inverse pole figure/image quality map. While many of these boundaries appear to occur at fairly high twist angles (~30°, as in Figures 6.21 (a-b) and ~20° in Figure 6.21 (c)), some have been observed at lower twist angles (~13° in Figure 6.21 (d)). It is interesting to note that in these particles, the cracks appear to select boundaries, which are nearly parallel to the basal planes. An analysis of 43 microcracks observed near fracture surfaces of eight different samples from this dataset clearly showed that 25 microcracks (~60%) were of a basal twist boundary, and one was a combination of basal twist/tilt. The majority of these (17 of 25) were less than 10° twist
from the symmetrical 30° (e.g., 25°-30°) position between the [11\̅20] and [10\̅10]. A histogram of the data, shown in Figure 6.22, clearly demonstrate that large number of microcracks, occur when the basal twist is closer to the symmetric position. This fraction of microcracks occurring along such special boundaries is significant, however the total fraction of such boundaries in the bulk specimens is determined to be ~1.8% to 3.6%. The fraction of these special boundaries are determined from the large scans of undeformed material from three specimens and calculated as a ratio of the lengths of special boundaries to length of total boundaries. This data supports the expectation that there is no correlation between alloy content and fraction of such special boundaries. The details of the 43 microcracks associated with equiaxed α particles are shown in Table 3, and the lengths of special boundaries and total boundaries in bulk scan are shown in Table 4. Of additional interest is the fact that of the 17 microcracks that did not appear to originate from a basal twist, 7 (~16%) contained colony boundaries. An additional analysis of one region containing two microcracks indicated the presence of a third basal twist boundary (see boundary #3, Figure 6.23), which did not crack. The degree of coplanarity between the interface and the basal planes was estimated from the trace of the cracks and the trace of the basal planes. The results indicate that the two boundaries which did initiate microcracks are oriented such that their traces are within 2° of the basal traces of the adjacent grains. However, the one boundary which did not initiate a microcrack is oriented such that its trace is ~ 14° from the basal traces of the adjacent grains. Therefore, not only is the relative orientation of the two adjacent equiaxed alpha
particles important, but also the degree to which the boundary is parallel to the basal plane.

**Additional Characterization of microcracks**

There also appears to be a rather significant difference in the degree of microcracking present in samples of varying toughness, with a larger number of cracks observed in samples with a lower toughness. Therefore, it is likely that in these α+β-processed Ti-6Al-4V systems the microcracking occurs ahead of the crack tip in the high stress field with the stress state resulting in plasticity and interfacial cracking, rather than behaving as an extrinsic toughening mechanism as occurs in some systems. When considering the average feature size (<15 µm) and the size of the plastic zone (calculated to be 180 µm to 1130 µm using the plane strain plastic zone size calculation, appropriate for all centerline observations), the distance from the fracture surface that such microcracking occurs (~100 µm) provides additional support for the theory that such cracking occurs in the high stress/strain fields ahead of the crack tip.

To further explore some of the features associated with such microcracking, the DualBeam FIB/SEM with an in-situ OmniProbe micromanipulator was used to extract a volume of material containing such a microcrack from a polished cross section and weld it onto a pillar on an OmniProbe TEM sample grid. The DualBeam FIB/SEM was then operated in a Slice-and-View™ mode to serially section through the material, successively removing small portions of material and imaging the FIB-prepared surface using an SEM column. Figure 6.24 (a-h) show selected SEM micrographs of the microcrack. In these micrographs, the material that is the subject of observation appears
as a four-sided polygon. The beta phase is the lighter gray phase, while the alpha phase is
darker. The vertical lines which appear are a FIB artifact called “curtaining” which can occur if the beam is influenced by a surface variability – in this case, the crack itself. As the curtaining occurs after interacting with the crack, it does not influence the interpretations. The first image to show any portion of the microcrack is shown in Figure 6.24(a). Here, there is a small visible defect in the material just above the light gray beta rib. Within 200nm, the crack appears more fully developed (see Figure 6.24(b)). It is interesting to compare Figure 6.24 (b and c) and observe that the beta rib, which, clearly delineate the neighboring alpha particles (Figure 6.24 (b)), is no longer observabed (Figure 24(c)), yet fundamentally, there still must exist a type of boundary, which the crack continues to follow. Figure 6.24 (e) contains not only the main crack, but a defect appearing as a “pore” in 2-D between the crack tip and the beta rib. Figure 6.24 (f) and 6.24(g) show the beta appearing to arrest the crack tip. The presence of the microvoids can be seen in Figure 6.24 (h).

6.4.4 Dislocation Analysis of Microcracks

The examination of a longitudinal cross section of the fracture surface suggested that a majority of microcracks is present between the boundaries of equiaxed alpha particles. The nature of the grains surrounding these microcracks is explored by electron back scattered diffractions (EBSD). The important outcome of this work suggested that majorities of microcracks are present at basal twist boundaries. It is also important to note that the majority of these microcracks are parallel to the traces of the basal planes of the
grains. The microcracks are present at equiaxed alpha particle boundaries irrespective of their orientation to the loading direction.

Given the nature of these microcracks at boundaries of equiaxed alpha particles, it is important to study the types of slip systems activated during the loading. The important slip systems in the HCP $\alpha$ phase are listed in Table 6.3. These are basal slip, prism slip, and pyramidal slip with the $<a>$ and $<c+a>$ Burgers vector. The primary slip system in the HCP $\alpha$ phase is prism slip. The Schmid factor analysis of the equiaxed alpha particles can show what types of slip systems are activated under loading. This section involves a systematic approach of identifying these boundaries with a microcrack, performing Schmid factor analysis, undertaking site-specific TEM sample preparation at the microcracks and subsequent dislocation analysis of these prepared foils.

Figure 6.25 shows a back scattered electron micrograph of the longitudinal cross section of the fracture surface of a $\alpha+\beta$ processed sample. It can be seen that the presence of microcracks in the cluster of equiaxed alpha particles. These microcracks are labeled in Figure 6.25. The orientations of the equiaxed alpha particles surrounding these subsurface microcracks were further examined using EBSD. Figure 6.26 shows an inverse pole figure map laid over with an image quality map which provides a knowledge of the orientations of different grains. The grains are color-coded based on the orientation. The misorientation between the two grains in microcrack #1 is $15.4^\circ$ [0001]. This microcrack involves a special orientation, which was discussed in the previous section of this chapter. The trace of the basal plane is parallel to the crack. Further investigation of the Schmid factor analysis was performed on the EBSD data. The Schmid factor map is
shown in Figure 6.27. It is important to note in Figure 6.27 that the Schmid factor is calculated for equivalent slip planes and directions. It is color coded based on the Schmid factor. The grains which were color coded red have the higher Schmid factor corresponding to that slip system while the grains color coded blue have lower Schmid factor. Presumably, the grain that has a high Schmid factor can slip easily for that slip system. It is clear from the Figure 6.27 that microcrack #1 has a higher Schmid factor for prism slip, pyramidal <a> slip and pyramidal <c+a> type slip.

Cross-sectional site specific TEM foils were prepared to examine these slip systems in the TEM. The FIB cross sectional foil prepared at microcrack #1 in Figure 6.25 is shown in Figure 6.28. In this figure, the left side of the microcrack corresponds to the top grain and the right side of the crack corresponds to the bottom grain as shown in Figure 6.25. Dislocation analysis in the equiaxed alpha particles suggests extensive slip on the basal plane. Figure 6.29 shows the basal slip in equiaxed alpha particles of the various samples. The trace of the basal planes clearly observed from these BF images. Figure 6.29 (a) is corresponds to the foil prepared from the microrack in Figure 6.25. Figure 6.30 shows a BF image consisted with dislocations emitting from crack tip. Basal slip and pyramidal <c+a> type slip can also be noted. Extensive TEM analysis was performed in the foil prepared near the crack tip to elucidate the deformation substructures. The presence of a very high dislocation density near the crack indicates that the nature of deformation is very complex. However, careful contrast experiments performed in the TEM were able to reveal the nature of deformation substructures.
The deformation near the crack tip is mostly planar. This planarity of deformation in Ti alloys helped to determine the major glide planes relatively easily although the exact nature of dislocations on those glides planes required extensive contrast experiments in the TEM. For example, the BF TEM image shown in Figure 6.31, which was recorded with the zone axis [2-1-10] shows three intense slip traces labeled B, P1 and P2. Figure 6.32 shows the [2-1-10] zone axis pattern. These slip traces are straight and thin since the glide planes are imaged on edge near this zone axis. The glide planes are co-planar and hence parallel to the beam direction [2-1-10] zone axis. The two most intense slip traces B and P1 have been determined to be basal (0001) and pyramidal (01-1-1) planes respectively and the less intense slip trace P2 has been identified as the pyramidal (01-11) plane.

In order to determine the character of dislocations (Burgers vector, \(<b>\)) on these slip traces, the sample was tilted away from their edge orientations to reveal the dislocation content on the glide planes. Figure 6.33 clearly reveals the long segments of dislocations running parallel to the slip trace that lie on the basal plane. These are \(<a>\)-type dislocations. Contrast experiments have shown that these are \(<a>\) dislocation with Burgers vector, \(b = 1/3[1-210]\). These dislocations are visible with \(g-[1-101], g-[1-100], g-[1-101], g-[01-11], g-[01-10]\) and \(g-[0-111]\) and invisible with \(g-[0002]\). The long segments of these dislocations are screw in character.

Similarly, the dislocations within the pyramidal slip trace P1 were imaged in Figure 6.34 after tilting away from their edge orientation shown in Figure 6.31. Contrast experiments have shown that these are \(<a>\) dislocation with Burgers vector, \(b = 1/3[2-1-}
These dislocations are visible with $\mathbf{g}$-[1-101], $\mathbf{g}$-[1-1-00], $\mathbf{g}$-[1-101], and invisible with $\mathbf{g}$-[01-11], $\mathbf{g}$-[01-10], $\mathbf{g}$-[0-111] and $\mathbf{g}$-[0002].

In contrast, the dislocations within the slip trace P2 (see Figure 6.35) follow the same trend as the dislocations within the trace B and therefore they have the same Burgers vector, i.e $\mathbf{b} = 1/3[1-210]$. This is consistent with the possibility that these dislocations can cross slip between the (0001) basal and ((01-11) pyramidal plane. TEM image shown in Figure 3 shows such a cross slip process.

In addition to the $<\mathbf{a}>$ slip described above the region near the crack tip has also undergone $<\mathbf{c}+\mathbf{a}>$ slip. The $<\mathbf{c}+\mathbf{a}>$ activity can be seen in Figure 3 imaged with $\mathbf{g}$-[0002] where all $<\mathbf{a}>$ type dislocations are out of contrast.

In addition, substructures resembling subgrains with less than 2-3 degrees misorientation are seen near the crack tip. The two beam BF TEM image shown in Figure 6.36 shows such substructures. It is speculated that the severe deformation caused by the intense stress conditions near the crack tip might have been the reason for the formation of these substructures.

### 6.5 Conclusions

An integrated Bayesian neural network modeling and critical experimental approach has been used to isolate the effects of composition and microstructure on the fracture toughness of $\alpha+\beta$ processed Ti-6Al-4V. The salient conclusions from the modeling include the following.
(1) It is possible to differentiate the continuum and mechanistic aspects of fracture toughness. At a continuum level, it is clear that the plane strain thickness (e.g., sample thickness) and yield strength play dominant roles. It is also clear from the virtual experiments for models including and excluding yield strength that the thickness of the alpha laths and the volume fraction of the equiaxed alpha particles, while directly influencing the yield strength of the material (and hence its resistance to the crack-tip opening), do not directly influence the toughness through other mechanistic means.

(2) It is possible to estimate the effect of plane strain thickness of toughness values that do not satisfy the strict $K_{1C}$ requirements. Indeed, $K_Q$ may be as much as 10% greater than a corresponding $K_{1C}$ would be. This estimation represents a powerful use of the neural network approach.

(3) The size of the equiaxed alpha particles is the dominant microstructural feature for microstructures, which fall within a certain range of microstructures. Subsequent characterization, guided by the modeling, has identified a certain type of basal twist boundary as playing a key role. This boundary, occupying less than 4% of total boundary length, can be linked with ~60% of microcracking events. In addition, it has been shown that the interface plane is important, with microcracking occurring when the plane is nearly parallel with the basal plane of both adjacent grains. The
second most common source of microcracking is colony boundaries (~17%).

(4) The basal twist boundaries may be enriched in β-stabilizers and depleted in α-stabilizers, possibly resulting in the stabilization of the β-phase for sufficiently enriched alloys. This would explain the observation that in small amounts (<0.41 wt%), Fe increases both yield strength and toughness.

The use of this combined approach has yielded many new insights into the very complex problem of fracture toughness. Indeed, such interpretations are rarely made on very ductile metallic systems such as α+β processed Ti-6Al-4V. Thus, an ulterior consequence of this approach may be its application in the development of mechanistic understandings for similarly complex properties in multi-phase ductile systems.
Table 6.1 The composition, microstructure and tensile properties of samples shown in Figure 6.7

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<th>Yield Strength (Mpa)</th>
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Table 6.2 Quality of different models

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Table 6.3 Slip systems in the HCP α phase
Table 6.4 g.b analysis of dislocations in HCP α phase shown in Figure 6.35

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Figure 6.1 Schematic of the complex nature involved in deriving fundamental details of the fracture toughness problem.

Figure 6.2 Inverse relationship between the fracture toughness and YS for various Ti alloys [55]
Figure 6.3 Effect of sample thickness on fracture toughness [58]

Figure 6.4 Schematic of the different generations of NN models
Figure 6.5 Predicted YS for fracture toughness database

Figure 6.6 Plane strain thickness measurements from fractured sample
Figure 6.7 BSE images of the sample that exhibit nominally the same KQ
Figure 6.8 NN models (a) YS, RA, PS thickness, (b) Composition and microstructure (c) Composition, microstructure and YS (d) Composition, YS, microstructure and PS thickness
Figure 6.9 Effect of YS on KQ as determined by model, which include (a) Composition-Microstructure-YS (b) Composition-Microstructure-YS-RA (c) Composition-Microstructure-YS-PS thickness

Figure 6.10 The functional dependence of plane strain thickness on KQ when YS included in the model
Figure 6.11 Trend plot of KQ when YS is excluded as one of the inputs (a) functional dependency on VF equiaxed α (b) Size of the equiaxed α (c) α lath thickness.

Figure 6.12 Trend plots of KQ when YS is included as one of the inputs (a) VF of equiaxed α (b) Size of the equiaxed α (c) α lath thickness.
Figure 6.13 (a-d) Influence of the composition on $K_Q$ when YS is excluded in neural network model
Figure 6.14 Influence of composition on KQ when YS is included in the NN model
Figure 6.15 Cross section of the fracture surface

Figure 6.16 Relation of (a) size of equiaxed alpha in bulk, (b) VF of equiaxed alpha to VF equiaxed occupied by fracture surface
Figure 6.17 Relation between the ratio of calculated surface area and particle volume fraction on Fv occupied by equiaxed alpha

Figure 6.18 Subsurface microcracks on the particle boundaries
Figure 6.19 Electron micrographs showing microvoid coalescence
Figure 6.20 Micrographs showing subsurface cracks (a) Inverse pole figure (b) Image quality map
Figure 6.21 EBSD showing basal on basal predominance (a) ~30° twist boundary along [0001] (b) ~30° twist along [0001] (c) ~21° twist along [0001] (d) ~13° twist along [0001]
Figure 6.22 Probability of occurrences of basal twists across the equiaxed α particles

Figure 6.23 Occurrences of basal twist boundaries with microcracks or without microcrack
Figure 6.24 Section through microcrack showing void nucleation
Figure 6.25 BSE image showing microcracks at equiaxed α particles -sample ID 89-3

Figure 6.26 IPF map of figure 6.25 (a) loading direction is in the y-direction of the page  
(b) IPF map when rotated 90 w.r.t TD-loading direction is normal to the page
Figure 6.27 Schmid factors map for different slip systems in the $\alpha$ phase. Blue color indicates the low Schmid factor and red color represent highest Schmid factor. (a) basal $\langle a \rangle$ slip (b) prism $\langle a \rangle$ slip (c) pyramidal $\langle a \rangle$ slip and (d) pyramidal $\langle c+a \rangle$ slip
Figure 6.28 Cross sectional TEM foil prepared from microcrack #1 in Figure 6.25 shows microcrack contained in the foil.

Figure 6.29 TEM bright field images showing extensive basal slip in equiaxed alpha particles. Bottom two images came from foils of different microcracks.
Figure 6.30 BF TEM image showing dislocations emitting from the crack tip
Figure 6.31 BF image showing basal slip and pyramidal slip

Figure 6.32 SAD pattern-Zone axis $[2 -1 -1 0]$
Figure 6.33 BF image showing basal slip

Figure 6.34 BF image showing pyramidal slip (P1)
Figure 6.35 Two beam BF image showing pyramidal slip trace (P2). P2 is cross slip onto basal plane

Figure 6.36 Two beam BF shows subgrains near the crack tip
Chapter 7 Neural Network Modeling of Tensile Properties of $\beta$ Annealed $\alpha+\beta$
Titanium alloys

7.1 Abstract

Modeling of the interrelationship between microstructure and mechanical properties of fully lamellar microstructure in $\alpha/\beta$ Ti alloys is rather difficult due to their complex microstructure with a wide range in length scale features. Non linear data modeling tools such as neural networks with Bayesian framework are implemented for these alloys to predict tensile properties. The database is comprised of composition and various microstructural features as inputs and tensile properties as outputs. Controlled experiments based on the neural network predictions have been designed to determine from the contribution of composition and individual microstructural features to tensile properties. Based on these experiments, Al, O, V and Fe appeared to have a positive influence on yield strength while width of the $\alpha$ lamellae, $\alpha$ colony size are important variables that have a negative influence on YS.

7.2 Introduction

Alpha+beta Ti alloys with fully lamellar microstructures are typically used in applications where damage tolerance is a primary concern because these alloys have superior fracture toughness and crack propagation resistance than alloys with bimodal
microstructures of the same composition. The relative advantages of both bimodal and lamellar microstructures are given in Table 7.1. Lamellar microstructures in α+β Ti alloys are typically obtained by β-processing or β-annealing both of which are conducted above the β-transus temperature. Figure 7.1 shows an optical micrograph of the β-annealed sample. A typical lamellar microstructure comprises of grain boundary alpha decorated along the prior beta grain (PBG) and in the prior beta grain either α colonies or basketweave type alpha are observed depending on the cooling rate. Secondary α in the β-matrix may also be observed in some heat treatments. Here, α-colonies consist of a group of similarly oriented alpha lamellae, all of them belonging to one variant of alpha where as the basketweave type microstructure consist of α lamellae of different variants. Alpha lamellae in a colony are separated by thin films of the beta phase. The α phase in colonies typically obeys the Burgers orientation relationship, where close packed planes and directions of the low temperature α phase are parallel to the close packed planes and direction of high temperature β phase [15]. The Burgers orientation relationship has an important effect on microstructural evolution in these alloys. The above-mentioned microstructural features are sensitive to the alloy chemistry and various thermomechanical processing routes. The size of the prior beta grains is controlled by temperature and time above β transus temperature. The propensity for large colonies in these alloys is increased by presence of higher α stabilizers or lower β stabilizers, higher β solutionization temperature, time, decrease in amount of β-work and decrease in cooling rate [85].
Important microstructural features that effect mechanical properties in lamellar microstructures are prior \(\beta\) grain size, \(\alpha\)-colony size, % colony, width of the \(\alpha\)-lamellae and volume fraction of alpha. Effects of these important microstructural features on mechanical properties have been studied in literature [12, 20, 21, 73]. It is suggested that the size of \(\alpha\) colony and width of the \(\alpha\) lamellae influence yield strength. As the size of the \(\alpha\) colony increases, the yield strength of the alloy decreases. This is attributed to an increase in slip length because slip length scales with the size of the microstructural feature. The alloy with basketweave type microstructure has higher yield strength than the alloy with colony microstructure because of reduction in effective slip length. The grain boundary alpha thickness does not have much effect on yield strength but it does have effect on ductility. Plastic deformation preferentially occurs on continuous grain boundaries of alpha leading to crack nucleation. The effect of grain boundary alpha thickness on mechanical properties is more significant in high strength \(\beta\) Ti alloys than \(\alpha+\beta\) Ti alloys. It is suggested in the literature that typical lamellar microstructures have faceted fracture under monotonic or cyclic loading conditions. These facets are typically observed on basal planes. The propensity for the faceted fracture increases with the increase in colony size. Faceted fracture is attributed to the intense shear activity across the \(\alpha\)-lamellae in a \(\alpha\) colony under both monotonic and cyclic loading. This intense shear activity is attributed to the reduction in tensile ductility of \(\alpha+\beta\) Ti alloys with the lamellar microstructure [85].

The above section summarized the effect of important features of lamellar microstructures on mechanical properties, however qualitatively. The development of
physics based models that relate microstructure and mechanical properties quantitatively is difficult because the microstructural features are complex and interdependent. For example the size of the $\alpha$-colony depends on the size of the prior $\beta$ grain. It is difficult to alter these features independently. So given the difficulties associated with lamellar microstructures of $\alpha+\beta$ Ti alloys, it is important to develop rules based models such as neural networks. This chapter demonstrates the application of neural networks to study the microstructure and mechanical property interrelationships in $\beta$-annealed $\alpha+\beta$ Ti alloys.

7.3 Experimental Techniques

The nominal composition of the alloy used in this work is Ti-xAl-yV ($4.76 < x < 6.55$, $3.4 < y < 4.5$) with controlled additions of O and Fe. Table 7.2 the shows chemical analysis of the alloy system in the current database and different grades of Timetal 6-4. It is clear from the Table 7.2 that composition of the alloys in the current database is within composition range allowed in Ti-6Al-4V. A total of nine alloys, each having a different composition within the above mentioned composition ranges are prepared. These alloys are solution treated in the $\beta$ phase field and cooled through the $\alpha+\beta$ phase field to room temperature under different cooling rates. The targeted cooling rates are 50 $^{0}\text{F}/\text{Sec}$, 150 $^{0}\text{F}/\text{Sec}$, and 400 $^{0}\text{F}/\text{Sec}$. But the measured cooling rates are $\sim 50 ^{0}\text{F}/\text{Sec}$, $\sim 140 ^{0}\text{F}/\text{Sec}$ and 250 $^{0}\text{F}/\text{Sec}$. This heat treatment schedule is shown in Table 7.3. The heat-treated samples were sent to MetCut Inc to perform the tensile and fracture toughness tests. The samples for the mechanical testing are chosen such that they are symmetric about the radius of the billet to minimize the texture effects. A mechanical property database is developed from
the results of these tests. A quantitative microstructural database is developed from the metallographic study of the grip section of the tensile and fracture toughness samples. The complete details of the microstructural characterization and database development in these alloys are explained in experimental section of this dissertation.

7.4 Results and Discussions

As has already been learned, that the important alloying elements are Al, V and controlled additions of O and Fe. Figure 7.2 shows the scatter plots of the yield strength on composition. The data in the compositional input space is not uniform. The data is noisy in some regions of the compositional input space while data is lean in some regions of the input space. This is shown in Figure 7.2 where data at lower and higher limits of compositional space is noisy and in the middle of compositional space is sparse. However, careful observation of Figure 7.2 shows that Al and O have a positive influence on yield strength, while V and Fe does not show any trend. Even though the effect of O and Al show certain trends, the data is scattered. A range in yield strength values can be obtained for certain O compositions. This is also true with other alloying elements. However, this range in yield strength may be due to other input variable are also changing. The effect of composition on ultimate tensile strength (UTS) is shown in Figure 7.3 and the trends of UTS on composition are similar to those trends of YS. The effect of composition on reduction of area (RA) is shown in Figure 7.4. It is clear from this figure that Al decreases the RA while the remaining alloying elements do not show any trend. This lack of trend with composition is most probably due to other input variables also changing simultaneously.
Based on legacy understanding, the important microstructural features in lamellar microstructure are width of the $\alpha$ lamellae, size of the $\alpha$ colony, prior beta grain size, volume fraction of alpha and volume fraction of $\alpha$ colony. These microstructural features are quantified using rigorous stereological procedures and their effect on tensile properties is shown through a series of scatter plots. Figure 7.5 shows the scatter plot of YS on important microstructural features. It is clear from this figure that the microstructural input space in the database is also not uniform. There are regions in the database where the microstructural input space is sparse in data and in some regions the data is noisy. Figure 7.5 (a) shows the data is noisy at 100% $\alpha$ colony. It is difficult to analyze data and visualize any trends between microstructure and tensile property through linear regression because these microstructural features are complicated and vary interdependent fashion. Neural networks are employed to determine the quantitative understanding of these inputs on mechanical properties.

### 7.4.1 Prediction of Yield Strength of $\alpha+\beta$ Ti Alloys with Lamellar Microstructure

In this work, the Bayesian framework developed by Dr. Mackay [63] is used to develop neural networks. The Bayesian framework is used because the uncertainty in the model prediction is calculated and also prevents over fitting. This uncertainty is calculated in terms of error bars in the model prediction. These error bars are especially useful in regions of input space where the data is noisy and sparse. The database that is required for the implementation of the neural networks is populated with composition, microstructural features and tensile properties. Here, composition and various microstructural features are used as inputs while tensile properties are used as outputs.
The distribution of the composition in the current database along with various grades of Ti-6Al-4V are shown in Table 7.2 while the distribution of microstructural features is shown in Table 7.4. The mechanical properties measured for the database are shown in Table 7.5. There are 52 samples in the database with four compositional inputs and five microstructural inputs making 468 data points in the database. Out of 52 samples, the database is divided into a training set consist of 38 samples and a test set consist of 14 samples. Figure 7.7 shows predictions of yield strength based on the best model. It is evident from the Figure 7.7 that the predicted yield strength values are very well in agreement with the experimentally measured yield strength values. The model predicted yield strength values with a precision in which errors are less than 5% experimental yield strength values. The correlation coefficient, R-value, which measures how well the experimental values are correlated to the predicted values, is obtained from the curve fitting of the predictions for different models. The values of these coefficients are shown in Table 7.6. The values of R which are close ~1 suggest the strong correlation between the experimental yield strength and predicted strength from neural networks.

7.4.2 Development of Neural Networks Models for Ultimate Tensile Strength (UTS)

The procedure for developing neural network to predict the UTS is similar to the development of models for the YS. The compositional inputs are Al, V, Fe and Oxygen while microstructural inputs are α-colony size, width of the α-lamellae, PBGF, and volume fraction of total alpha. Various models have been developed with different seeds and hidden nodes and models are ranked in terms of test errors. These models can be used to make predictions and predications from such model are shown in Figure 7.8. It is
clear from the Figure 7.8 that the predicted UTS values are very well matched with experimental UTS values in both the training and test set. The quality of this model prediction is evaluated with a predefined range in error values as a guide. In Figure 7.8, two straight lines that bound predictions correspond to ± 5 % error. Other than a small number of data points, whose prediction are deviated more than 5 %, the majority of the samples whose predictions are well below the 5 % error. In this case a committee model, which is an average of best two models is used to make UTS predictions. Because sometimes the predictions from a single best neural network model are not sufficiently accurate, then it is necessary to use several model together to make reliable predictions. The use of committee model does not affect the predictions in an input space where model has good fit. It only improves the reliability of the predictions in an input space where there is an uncertainty in the fit. The committee model gives optimum predictions because each model in the committee gives importance to different inputs. The data analysis on the predicted values is shown in Table 7.7. Two important points emerge from the Table 7.7, first, the values of correlation coefficient, R is close to 1 for both training and test set showing how closely the predicted values are with experimental values; Second, the improvement in the R value of the predictions in a test set of committee model shows the improvement in model predictions.

The models related to yield strength or UTS are helpful in predicting the mechanical properties, however it is necessary to understand the effect of the individual microstructural features and composition on mechanical properties. The development of
virtual experiments can provide an estimate of contributions from each individual variable and the methodology of doing this is discussed in the following section.

7.4.3 Development of Functional Dependencies of YS and UTS on Composition and Microstructural Features

The neural networks have ability to study the significance of each input to certain mechanical property. The relevance of each input influencing the yield strength for certain models is shown in Figure 7.9. This figure shows the how a neural network model gives significance to importance to composition and microstructural features that affect the yield strength or ultimate tensile strength. It is clear from the Figure 7.9 that different models assign significance to different inputs. This suggests the importance of making predictions not only from the best model but also from the several best models and comparing their predictions. From Figure 7.9 it is follows that Al, Fe and O are pertinent in influencing the yield strength while volume fraction of colony, CSF, PBGF and VF total alpha are important microstructural variables that significantly vary yield strength. A Similar study was undertaken to reveal the relevance of each input on influencing the UTS and it is shown in Figure 7.10. The perceived significance to different inputs is shown for different NN models. It is clear from the Figure 7.10 that Al, Fe and O are important inputs related to composition and lath thickness and PBGF are important microstructural inputs. It is important to note that these can only suggest how important are inputs influencing the mechanical properties but they do not give any quantitative information.
In order to study effect of certain inputs on mechanical property, one would require developing virtual experiments where the effect of particular inputs on outputs is studied when that input is systematically varied while the remaining input variables in the database are kept at an average value. The predictions of yield strength from the virtual experiments on composition and microstructural features are shown in Figure 7.11 and 7.12 respectively. In these figures functional dependencies from the two best models are plotted. These models predict the same trend of YS on composition and microstructure but the predictions may vary to a certain extent. The error bars on the predictions are calculated with one standard deviation. From the virtual experiments it is determined that alpha stabilizers such as Al and O increase the yield strength significantly and this trend is consistent with the effect of Al and O have on YS of α+β processed Ti alloys. Fe is an important β stabilizer that affects yield strength while V does not seem to affect yield strength significantly. The effect Fe on yield strength is significant considering it is only coming from the 15 % of the sample volume i.e., the β phase. The effect of composition on yield strength is quantified per wt% and listed in the Table 7.10. It can be seen from the Table 7.9 that 1 wt% Al, V, Fe and O increases the yield strength by ~50MPa, 12~MPa, 80 MPa and 1000MPa respectively. These values are consistent with the predictions from the yield strength of α+β processed α+β Ti alloys. This trend is surprising because β-annealed and α+β processed Ti alloys have different microstructures and yet they have similar effect of composition on yield strength. This leads to a conclusion that the effect of composition on yield strength is consistent with
the solid solution strengthening. The increase in O content in the alloys could promote the planar slip thus increasing the yield strength.

The effect of microstructural features on yield strength is shown in Figure 7.12. The effect of $\alpha$ colony size is shown in Figure 7.12 (a). As the size of the $\alpha$ colony increases, the yield strength appears to decrease. This trend is consistent with observations from the literature. The decrease in yield strength with increase in colony size can be explained in terms of slip length argument. The slip length is proportional to the colony size and legacy data suggests an inverse relationship between slip length and yield strength.

The effect of prior $\beta$ grain size on yield strength is shown in Figure 7.12 (b). Prior $\beta$ grain factor, which is a measure of prior $\beta$ grain size, is defined as area per unit volume. The larger the PBGF number, the smaller the prior $\beta$ grain size. From Figure 7.12 (b) it is evident that the yield strength decreases with increase in prior $\beta$ grain size. This is also consistent with the literature, however this effect is quantitatively shown here. The size of the prior $\beta$ grain limits the size of the colony. The smaller the $\beta$ grain size then smaller is the $\alpha$ colony. The small beta grain reduces the effective slip length and causes an increase in yield strength.

The effect of volume fraction of alpha on yield strength is shown in Figure 7.12 (c). The $\alpha$ phase is relatively stronger than the $\beta$ because of availability of limited slip systems. This trend is also consistent with the literature. The effect of the % colony is shown in Figure 7.12 (d). The yield strength initially increases with the % colony and decrease with further increase in % colony in the microstructure. The overall trend shows
decrease in yield strength with the increase in % colony in the microstructure. The decrease in yield strength is understood because when the microstructure transforms from basketweave to a colony type microstructure, the effective slip length is increased. This increase in slip length causes a reduction in yield strength values. But the initial increase in yield strength (Figure 7.12) is yet to be understood. The effect of α lath width on yield strength is shown in Figure 7.13. The yield strength decreases with increase in α lath width. This trend is also consistent with the legacy data. The reduction in yield strength is expected with an increase in α lath thickness because slip length is proportional to the lath width and inversely scale with the yield strength.

The control experiments are also performed for UTS to see the extent of influence of composition and microstructural features on UTS. The trend plots of UTS on composition are shown in Figure 7.14. The effect of composition on UTS is similar to the effect to the effect of composition on YS. The magnitude of the influence of each alloying element on UTS composition is given in Table 7.9. Al, O and Fe are important alloying elements influencing UTS. The effect of microstructural features on UTS is shown in Figure 7.15. Again, trends of UTS on microstructural features are similar to the trends observed for YS with microstructure. The ultimate tensile strength of the β-annealed Ti alloys is inversely scale with α colony size and width of the α lamellae and positively scale with the volume fraction of alpha. The effect of prior β grain size on UTS is not consistent with its effect on YS. The exact mechanism for this trend is not well understood.
7.4.4 Prediction of Uncertainty in the Neural Network Models

The error bars on the trend plots of YS and UTS suggest uncertainty in the model predictions. These error bars show the reliability of the model predictions. The error bar becomes large when the data in input space is noisy or sparse. These error bars become obvious when the functional dependencies YS and UTS on composition and microstructural features are noted. For example at extreme values of Al and O, the error bars become large suggesting noise in the regions of that input space (see scatter plot YS, UTS on composition). The large error bars on functional dependency of UTS on CSF are due to the limited data in the regions of the input space (see scatter plots of YS and UTS on microstructural features). The error bars also become large when we use a committee model, which is an average of several best models. These committee models do not play a role where the fit is good but only effect the where the fit is not reliable. This is clearly shown in Figure 7.16, which shows the predictions from each individual model and the committee model of those individual models. The error bars become large in certain regions where the predictions from different individual models defer. Even though the predictions have large error bars, these committee models are some time helpful because they improve the reliability of the model predictions.

7.5 Conclusions

(i) The neural network modeling with a Bayesian framework is successfully implemented in predicting the tensile properties of β-annealed α+β Ti alloys
(ii) The interrelationship between the microstructure and tensile properties have been predicted and they are consistent with the legacy understanding.

(iii) Based on the virtual experiments Al, O and Fe are the important alloying elements and α-colony size, width of the α lamellae and prior β grain size are the important microstructural variables that effect tensile properties in these alloys.
Equiaxed Alpha:
- Higher Strength (for equivalent heat treatment)
- Higher ductility and formability
- Better low cycle fatigue (initiation) properties
- Better hydrogen tolerance
- Higher threshold stress for hot-salt stress corrosion cracking

Acicular:
- Superior creep properties
- Higher fracture toughness (better damage tolerance)

Table 7.1 Comparison of mechanical properties between bimodal and lamellar microstructures of $\alpha+\beta$ Ti

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<th>Fe, wt%</th>
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Table 7.2 Chemical analysis of current database and different grades of Ti6Al4V
Table 7.3 Details of the thermomechanical treatment of (β – annealing) samples in the database

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<td>3.313</td>
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<td>234.88</td>
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<td>1765</td>
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Table 7.4 Quantified microstructural data in the tensile database

<table>
<thead>
<tr>
<th>Microstructure (β-annealed)</th>
<th>Min</th>
<th>Max</th>
</tr>
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<tr>
<td>Colony Scale Factor, µm</td>
<td>23.09</td>
<td>192.17</td>
</tr>
<tr>
<td>Width of α lamellae, µm</td>
<td>0.21</td>
<td>0.48</td>
</tr>
<tr>
<td>Prior Beta Grain Factor (PBGF), mm²/mm³</td>
<td>2.78</td>
<td>29.48</td>
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<tr>
<td>% Colony</td>
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<td>100</td>
</tr>
<tr>
<td>VF of total alpha</td>
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<td>0.93</td>
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</table>
Table 7.5 Mechanical property data in the database

<table>
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<th>Mechanical Property</th>
<th>Min</th>
<th>Max</th>
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<td>Yield Strength, MPa</td>
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<td>930</td>
</tr>
<tr>
<td>UTS, MPa</td>
<td>728</td>
<td>1031</td>
</tr>
<tr>
<td>% RA</td>
<td>12.7</td>
<td>36.1</td>
</tr>
<tr>
<td>% Elongation</td>
<td>7.2</td>
<td>16.9</td>
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</table>

Table 7.6 Data analysis of the best models (linear correlation coefficient (R) values from the curve fitting shows the quality of the model predictions of YS)

<table>
<thead>
<tr>
<th>Models</th>
<th>R from curve fitting (Training Set)</th>
<th>R from curve fitting (Test Set)</th>
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<td>0.98833</td>
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Table 7.7 Data analysis of the best models (linear correlation coefficient (R) values from the curve fitting shows the quality of the model predictions of UTS)
<table>
<thead>
<tr>
<th>Alloying elements</th>
<th>ΔYS, MPa (for 1 wt%)</th>
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</thead>
<tbody>
<tr>
<td>Al</td>
<td>50</td>
</tr>
<tr>
<td>V</td>
<td>12</td>
</tr>
<tr>
<td>Fe</td>
<td>80</td>
</tr>
<tr>
<td>Oxygen</td>
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</table>

Table 7.8 Effect of composition on YS (Increase in YS with an increment of 1 wt% in composition)

<table>
<thead>
<tr>
<th>Alloying element</th>
<th>ΔUTS/1 wt %, MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>50</td>
</tr>
<tr>
<td>V</td>
<td>13</td>
</tr>
<tr>
<td>Fe</td>
<td>130</td>
</tr>
<tr>
<td>Oxygen</td>
<td>1030</td>
</tr>
</tbody>
</table>

Table 7.9 Effect of composition on UTS (Increase in UTS with an increment of 1 wt% in composition)
Figure 7.1 Optical micrograph of the β-annealed sample showing important features
Figure 7.2 Scatter plots of YS with (a) Al (b) O (c) V and (d) Fe
Figure 7.3 Scatter of UTS with (a) Al (b) O (c) V and (d) Fe
Figure 7.4 Scatter plots showing the effect of composition on RA (ductility)
Figure 7.5 Scatter plots of YS on (a) % colony (b) prior beta grain factor (PBGF) (c) colony scale factor and (d) lath thickness
Figure 7.6 Scatter plot of UTS with (a) % colony (b) PBGF (c) CSF and (d) α lath thickness
Figure 7.7 Neural network model predictions of β-annealed YS

Figure 7.8 Neural network model predictions of β-annealed UTS
Figure 7.9 Relevance of each input influencing the YS for different NN models
Figure 7.10 Relevance of each input influencing the UTS for different NN models
Figure 7.11 Effect of composition on yield strength
Figure 7.12 Variation in yield strength with microstructural features
Figure 7.13 Variation of YS with lath thickness

Figure 7.14 Variation of UTS with composition
Figure 7.15 Variation of UTS with microstructural features
Figure 7.16 Variation of UTS with different models (a) O and (b) lath thickness
Chapter 8  Neural Network Modeling of Fracture Toughness of β Annealed α+β Titanium Alloys

8.1 Abstract

Interrelationships between microstructure and fracture toughness of β-annealed α+β Ti alloys are difficult to predict because the microstructure varies in a complex fashion and features of the microstructure are interdependent. In this research, neural networks with Bayesian framework are implemented to predict fracture toughness in β annealed α+β Ti alloys. Various models have been developed to optimize the predictions. The effect of microstructural features on fracture toughness is studied with the help of a series of virtual experiments.

8.2 Introduction

The widespread use of α+β Ti alloys in airframes and engines is due to their balance of properties. There is an increased need to predict properties especially related fracture toughness because these properties control the size of a component in the above-mentioned applications. The improvement in properties is paramount importance because it enables efficient design of structural components and result in weight and cost savings. It has been suggested that microstructure is a single most important variable that affect fracture toughness extensively while other factors considered are texture, modulus and environment [86]. The microstructure in α+β Ti alloys is sensitive to alloy composition
and various thermomechanical processing routes. Two important commercial processing routes in α+β Ti alloys are α+β processing to obtain bimodal microstructure and β-annealing to result in a lamellar microstructure. Lamellar microstructures show superior damage tolerance properties, improved creep and increased resistance to crack propagation than the bimodal microstructure of the same composition. The important microstructural features that effect fracture toughness are prior β grain size (PBG), grain boundary α thickness, α colony size and width of the α lamellae. The above-mentioned microstructural features span different length scales and are interdependent. The detailed descriptions of these features are explained in Chapter 2 of this thesis. The microstructure and fracture toughness property relationships in these alloys are complex and characterized only qualitatively. For example, it is suggested that in materials with lamellar microstructures, fast fracture occurs primarily along prior beta grain boundaries [86]. These boundaries also act as sites for crack deflection, which increases the fracture toughness. It is suggested that grain boundary alpha relieves stress when it encounters the crack. This will force the crack to propagate along the α+β interface [87]. Greenfield suggested that there should be an optimum balance between width of the α lamellae and spacing between the α lamellae. He argued that α lamellae should be thick enough to deflect the crack and short and close enough to change the crack path frequently. The fracture toughness increase with the width of the α lamellae and decrease with the spacing between the α lamellae [83]. It is clear from the above examples that microstructure influences fracture toughness in a complex fashion. It is difficult to develop a mechanistic model to predict the interrelationships between the microstructure
and fracture toughness quantitatively. Bayesian neural networks are used to study the interrelationship between the microstructure and mechanical properties. Neural networks can be considered as short-term alternatives until more robust physics based models are developed. In this chapter, neural network with Bayesian framework is implemented to quantitatively characterize the interrelationship between microstructure and fracture toughness in β-annealed α+β Ti alloys. The compositions of the alloys used in this study are Ti –xAl-yV (4.76 <x<6.55 and 3.3 <y< 4.5) with controlled additions of Fe and interstitial oxygen.

8.3 Experimental Techniques

A database, which comprise of composition, microstructural features and fracture toughness has been developed. A total of nine alloys that belong to different melts of the alloy Ti-6Al-4V with variations in impurities such as Fe and interstitial O are subjected to different heat treatments to produce a considerable variation in lamellar microstructure. The microstructural data is quantified using rigorous stereological methods. The fracture toughness tests were conducted according to ASTM E-399 standards and the sample geometry used is compact tension type. The thickness of the plane strain region, which is distance between the shear lips on fracture surface, is measured and it has been used as one of the input variables in the neural networks. Neural networks have been developed with various combinations of inputs and neural networks architectures. The database consists of 52 samples, out of which, 38 samples are used to train the network and 14 samples are used to measure the performance of the network. The total number of data points in the training set actually vary based number of inputs in an input layer. For
example, a model with eight inputs has 304 data points in the training set. A number of models developed with a combination of different inputs and a summary of these models is shown in Table 8.2. The best models are selected based on minimum error in the test set and these models are used to make predictions.

### 8.4 Results and Discussions

Figure 8.1 shows the scatter plot of fracture toughness, $K_Q$, on composition. The scatter plot of $K_Q$ on composition shows the data is noisy and scattered. The scatter in the fracture toughness for a specific alloying element suggests an underlying effect of other input variables on fracture toughness. It is difficult to obtain a trend between the fracture toughness and composition based on linear regression while other inputs are being varied. This can be dealt using neural networks because they can capture the interaction between input variables. The scatter plots of $K_Q$ on microstructure are shown in Figure 8.2. It is clear from scatter plots that thickness of $\alpha$ lath and thickness of grain boundary $\alpha$ seem to effect fracture toughness while size of the $\alpha$ colony and prior $\beta$ grain size do not show any trend. Figure 8.3 (a) shows the scatter plot of $K_Q$ on yield strength. Although one would expect an inverse relationship between the yield strength and fracture toughness, in the current database there is no visible trend between them. There is scatter in the values of fracture toughness for the same yield strength suggesting variation in microstructure. The scatter in fracture toughness data is consistent with legacy data. The scatter in the legacy data is shown in Figure 8.4 [39]. It is also important to note that the range in fracture toughness for corresponding yield strength is about $\sim20$ MPa-m$^{1/2}$ where as it is approximately 50 MPa-m$^{1/2}$ in $\alpha+\beta$ processed alloy. The thickness of the plane
strain region is proportional to the yield strength, which can be clearly seen from Figure 8.3 (b). It may be concluded from the Figure 8.3 (b) that the stress state in the plane strain region is proportional to the yield strength.

A neural network model has been developed with important microstructural features and plane strain thickness as input and $K_Q$ as output. The predictions from the best model are shown in Figure 8.5. It is clear from the Figure 8.5 that the majority of predictions from both the training set and the test set lie within $\pm 5\%$ of error. It is important to note that the quality of the model is not as good as the quality of the model for fracture toughness of $\alpha+\beta$ processed Ti-6Al-4V. This may be due to the complexity of the microstructure in $\beta$-heat treated condition. Apart from the complexity of microstructure, it is also possible that some important variables have been missed. The performance of the model was improved with addition of other relevant inputs such as composition. The committee model also improved the reliability of the predictions. The predictions from a committee model are shown in Figure 8.6. It can be seen that all predictions lie within $\pm 5\%$ of the error.

In order to study the effect of each microstructural feature or composition on fracture toughness, it is necessary to conduct control experiments in which one input variable is systematically changed while the remaining input variables are kept at an average value. Figure 8.7 shows the influence of various microstructural features on fracture toughness. Figure 8.7 (a) suggests that a decrease in fracture toughness occurs with an increase in colony size. The extent of influence of $\alpha$ colony size has on $K_Q$ is small. The effect of $\alpha$ colony size on $K_Q$ can be explained somewhat mechanistically.
Thus, as the size of the colony increases for a fixed beta grain size, the number of colony boundaries decreases causing a few deflections in the crack path. This reduction in crack length decreases the fracture toughness. This is shown schematically in Figure 8.9 [29]. In lamellar microstructures, the roughness of the crack front profile dominates fracture toughness. The fracture toughness increases with an increase in resistance to crack propagation.

The effect of prior β-grain size on fracture toughness is shown in Figure 8.7 (b). The prior beta grain size, which is defined as prior beta grain factor, is measured in terms of area per unit volume, so large PBGF number corresponds to the smaller beta grain size. The increase in prior β grain size increases the fracture toughness. As the size of β grain increases for a fixed colony size then the number of colony boundaries will grow. This increase in the number of colony boundaries in a beta grain increases the roughness of the crack front and contributes to an increase in fracture toughness. This is shown schematically in Figure 8.9. The increase in volume fraction of colony increases the fracture toughness. The roughness of the crack front profile increases with the increase in volume fraction of colony and the roughness of the crack front profile influence the fracture toughness.

The effect of grain boundary α thickness on fracture toughness has been identified and its effect on $K_Q$ is shown in Figure 8.7 (d). The grain boundary α thickness positively influences the fracture toughness. The grain boundary α can deflect the crack path thus increasing the resistance to crack propagation. This result is consistent with the legacy observations [87]. The effect of width of the α lamellae on fracture toughness is
shown in Figure 8.8. The fracture toughness increases with an increase in lath thickness. This result is consistent with the legacy data. It has been shown that the crack initiation resistance increases with an increase in $\alpha$ lath thickness [29]. It has been concluded that an increase in fracture toughness can be obtained when laths are thick enough to deflect the cracks and short enough to change crack path frequently [87].

The influence of the composition on $K_Q$ is shown in Figure 8.10 and Figure 8.11. The $\alpha$ stabilizers such as Al and O decrease the fracture toughness. These trends are consistent with the literature. The V does not appear to influence fracture toughness while Fe seems to increase the fracture toughness. The effect of plane strain thickness on $K_Q$ is shown in Figure 8.12. The magnitude of this effect is small compared to its effect on fracture toughness of $\alpha+\beta$ processed Ti-6Al-4V, however it follows a similar trend.

8.5 Conclusions

The fracture toughness of the $\beta$ heat treated Ti6Al4V has been predicted using neural networks. The interrelationships between the various microstructural features and fracture toughness have been established. Alloying additions such as Al and O have a negative influence on fracture toughness. The effect of V on $K_Q$ is not understood completely, while Fe seems increase the fracture toughness. The width of the $\alpha$ lamellae increases the fracture toughness. The $\alpha$ colony size decreases the fracture toughness. The prior beta grain size increases the fracture toughness.
### Table 8.1 Microstructure and fracture toughness database of β-annealed alloys

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<tr>
<th>Input/Output</th>
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<th>Max.</th>
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<tbody>
<tr>
<td>Al, wt%</td>
<td>4.760</td>
<td>6.550</td>
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<tr>
<td>V, wt%</td>
<td>3.297</td>
<td>4.450</td>
</tr>
<tr>
<td>Fe, wt%</td>
<td>0.107</td>
<td>0.407</td>
</tr>
<tr>
<td>O, wt%</td>
<td>0.071</td>
<td>0.198</td>
</tr>
<tr>
<td>Width of α lamellae, μm</td>
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<tr>
<td>CSF, μm</td>
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</tr>
<tr>
<td>PBGF, mm$^3$/mm$^3$</td>
<td>3.21</td>
<td>17.72</td>
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<td>VF alpha</td>
<td>0.782</td>
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<td>% Colony</td>
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<td>GB alpha thickness, μm</td>
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<tr>
<td>$K_{IC}$, Mpa-m$^{1/2}$</td>
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Table 8.2 Schematic showing the different neural network models

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<th>Model 3</th>
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Figure 8.1 Scatter plot of KQ versus composition

Figure 8.2 Scatter plot of KQ versus microstructural features
Figure 8.3 Scatter plot of (a) KQ versus YS and (b) PS thickness versus YS

Figure 8.4 Scatter in fracture toughness suggesting variation in microstructure [39]
Figure 8.5 Neural network prediction of fracture toughness in β-heat treated alloys

Figure 8.6 Predictions from a committee model when composition added as additional inputs
Figure 8.7 Influence of microstructural features on KQ (a) Colony size (b) Prior beta grain size (c) GB alpha thickness and (d) % Colony

Figure 8.8 Influence of lath thickness on KQ
Figure 8.9 The effect of colony size and $\beta$ grain size on fracture toughness [29]

Figure 8.10 The influence of $\alpha$ stabilizers on KQ (a) Al and (b) O
Figure 8.11 The influence of β-stabilizers alloying elements on fracture toughness

Figure 8.12 The effect plane strain thickness on KQ
Chapter 9 Summary and Conclusions

This research has involved the application of Bayesian neural network modeling to develop accurate predictive models for properties in $\alpha+\beta$ processed and $\beta$ annealed Ti-6Al-4V alloys. In addition to the development of a well-populated database that consists of both microstructural and compositional variables for the training of the neural network, this thesis has also presented the predictions of tensile properties and fracture toughness.

In the Chapter 5 the tensile properties of $\alpha+\beta$ processed $\alpha+\beta$ Ti alloys have been predicted. The experimentally measured values of yield strength and ultimate tensile strength are very well in agreement with the predicted values supporting the reliability of predictions from neural networks. Apart from developing neural network models functional dependencies of tensile properties on composition and microstructural feature were also determined. The outcome of the functional dependencies indicate that volume fraction and size of the equiaxed particles are the most important microstructural variables that influence strength. Likewise, Al, O and Fe are most important compositional variables. The use of these models also resulted in the identification of a potential V-O synergistic interaction, which is not yet completely understood but may lead to the formation of nanometer scaled $\alpha$ laths under certain conditions, resulting in an increase in the yield strength.
In Chapter 6, an integrated approach has taken in coupling Bayesian neural network modeling and critical experiments to isolate the effects of composition and microstructure on the fracture toughness of α+β processed Ti-6Al-4V. It is possible to differentiate the continuum and mechanistic aspects of fracture toughness. At a continuum level, the plane strain thickness (e.g., sample thickness) and yield strength play dominant roles. It is possible to estimate the effect of plane strain thickness on toughness values that do not qualify valid $K_{1C}$ requirements. Indeed, $K_Q$ may be as much as 10% greater than a corresponding $K_{1C}$ would be. This estimation represents a powerful use of the neural network approach.

The size of the equiaxed alpha grain is the dominant microstructural feature that mechanistically effects fracture toughness. Subsequent characterization, guided by the modeling, has resulted in identifying a certain type of basal twist boundary as a potential site for microcracks. In addition, it has been shown that the interface plane is important, with microcracking occurring when the plane is nearly parallel to the basal plane of both adjacent grains. The second most common source of microcracking is colony boundaries (~17%).

In Chapter 7 neural networks with a Bayesian framework has been successfully implemented in predicting the tensile properties of β-annealed α+β Ti alloy. The interrelationships between the microstructure and mechanical properties have been established and they are consistent with the legacy understanding. Based on the virtual experiments, the important microstructural features that influence the tensile properties are α-colony size, width of α lamellae and prior β grain size.
The virtual experiments suggested Al, O and Fe are the important alloying elements that influence the tensile properties. It is also important to note that the effect of composition on yield strength of the β-annealed Ti alloys is consistent with the effect of composition on α+β processed Ti-6Al-4V.

In Chapter 8, the fracture toughness of the β heat treated Ti-6Al-4V has been predicted using neural networks. The interrelationships between the various microstructural features and fracture toughness have been established. The alloying additions such as Al and O have a negative influence on fracture toughness. The effect of V on $K_Q$ is not understood completely while Fe seems to increase the fracture toughness. The width of the α lamellae and prior β grain size increases the fracture toughness while the size of the α colony decreases the fracture toughness.
References


56. Pandat.


60. software, T.O.


Appendix A: Measuring Fv holes/cracks away from fracture surface
Duplicate Layer (Layer 1)

Bi-Level threshold (~20)

Reject Features <100  (takes stuff out of bakelite area)

Change opacity, compare to background

Clean up edge of fracture, black region

Change opacity back

Duplicate Layer (Layer 2)

Duplicate Layer (Layer 3)

Reject features < 2000

Remove remaining large features with brush

Measure Fv – this will give a baseline of what amount of image is in mount material

Skeletonize

Thicken Skeleton

Fill Area above with white and below line (fracture surface) with black

Set opacity to 50%

Shift down by x number of pixels (to correspond to a certain distance) – select the whole image, use the Move tool (V) (right top tool – looks like an arrow) to shift it down while looking at the distance in pixels in your info guide

Deselect

Fill all area above (that is now “empty” (appears black) with white)

Copy and Merge Layers 2 and 3
Measure volume fraction black

Repeat from “Shift down” down.