Effect of Oxygen Equivalence on the Strength and Fracture Toughness of Ti-6Al-4V Forgings

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Bachelor of Science

By

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Abstract

Tensile testing and fracture toughness testing were conducted to establish a numerical relationship between interstitial content and performance in forged Ti-6Al-4V. The value of oxygen equivalence was used to represent the interstitial content by combining the weight percent of oxygen, carbon, and nitrogen. The correlation between oxygen equivalence and mechanical properties can be used to accurately predict the performance of forged parts. Samples of forged parts with varying levels of interstitial content were subjected to a recrystallization anneal at 75 F below the beta transus temperature to decrease microstructure variability across parts with a second anneal at 1300 F to relieve residual stress. There was an increase in tensile strength with oxygen equivalence, but the numerical correlation could not be found due to lack of fit. There was a high amount of variation within the data for compositions A and B. The variation in tensile strength for compositions C-F is unknown because only one sample was tested from each composition. Specimen direction (longitudinal vs. transverse) was found to be insignificant for tensile strength. Fracture toughness was on average 11.4 ksi*in^0.5 higher in the L-T direction as compared to the T-L direction. The numerical effect of oxygen equivalence on fracture toughness was inconclusive due to the small data set.

Key Words

Titanium alloy, Ti-6Al-4V, Materials engineering, Oxygen equivalence, Beta transus temperature, Interstitial elements, Annealing, Tensile strength, Fracture toughness, Yield strength, Microstructure, Metallography,

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1 Introduction

Titanium alloys are widely used because their high strength and low density make them ideal for many structural applications. Compared to other metal alloys, titanium alloys possess an appealing combination of high yield strength, fatigue strength, temperature capability, and corrosion resistance [1]. Common parts made from titanium include wing fittings, frames, fan blades, and landing gear. Although titanium already has excellent mechanical properties, engineers are still working to improve them. Strength and fracture toughness are particularly important to optimize in structural applications.

Figure 1: A titanium landing gear beam made of Ti-6Al-4V, manufactured by Weber Metals.

1.1 Microstructure

Pure titanium has a body centered cubic structure, called the beta phase, at high temperatures and a hexagonal close packed structure, called alpha phase, at lower temperatures. The alpha phase is considerably less ductile and harder than the beta phase. Titanium is classified by the phases present at room temperature: alpha, beta or alpha+beta. Alpha+ beta alloys with less than 10% beta are called near alpha alloys. Ti-6Al-4V is an alpha + beta titanium alloy that contains 6 wt% aluminum and 4 wt% vanadium. Maintaining an alpha + beta microstructure at room temperature requires the addition of phase stabilizers [1]. Aluminum increases the temperature at which the alpha phase is stable. Vanadium allows the beta phase to be present at lower temperatures than the beta transus (Figure 2).

The beta transus temperature is defined as the lowest temperature at which the material is entirely beta phase, typically between 1750-1850°F.

$$BT = 1627.43 + 39.46*Al + 216*O + 1286*N + 873.875*C - 23.375*V - 44.365*Fe \qquad \text{Eq. 1}$$

Aluminum (Al), oxygen (O), nitrogen (N), carbon (C), vanadium (V), and iron (Fe) are expressed in weight percent. Beta transus temperature is in degrees Fahrenheit.

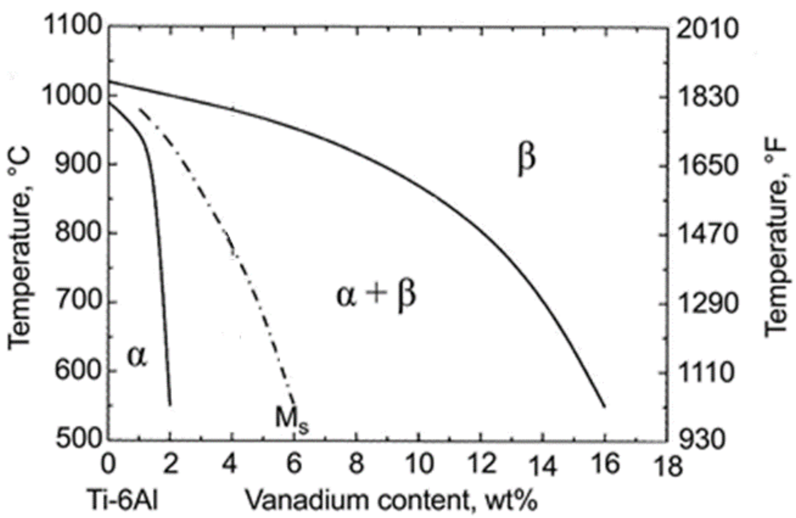


Figure 2: The beta transus temperature decreases as the vanadium content increases [2].

There are a variety of possible heat treatments for alpha + beta alloys which result in different microstructures. By changing the microstructure of the titanium, the mechanical properties can also be changed. Performing a heat treatment on alpha titanium does not have significant results because it does not go through a phase change. Alpha + beta titanium is the most widely used because it strikes a good balance between the properties of alpha and beta individually and because these properties can be improved with heat treatment.

Forging and subsequent heat treatment can result in the microstructure seen in Figure 3. The light regions are equiaxed alpha phase particles, while the dark regions are the transformed beta phase matrix. The grain size is affected by the cooling rate. At higher cooling rates the alpha grains will be smaller which tends to increase strength. At sufficiently high cooling rates a martensitic transformation can even occur.

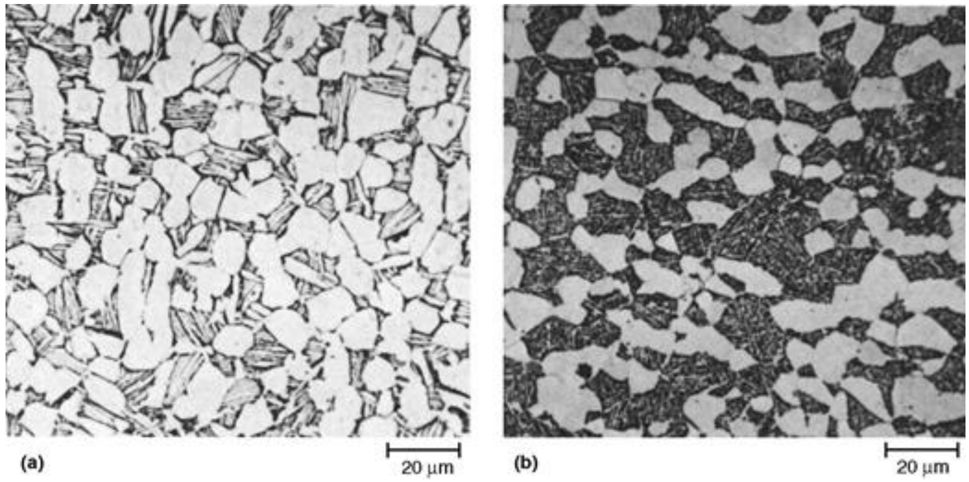


Figure 3: Ti-6Al-4V forging. (a) Solution treated 1 h at 955 °C (1750 °F), air cooled, and annealed 2 h at 705 °C (1300 °F). (b) Same as in (a), except water quenched from the solution treatment (before the anneal) instead of air cooled [3].

1.2 Mechanical Properties

Solute elements are often incorporated into alloys to produce a strengthening effect by impeding dislocation motion. This mechanism is due to the interaction between the lattice strain effects of dislocations and solute atoms. If the solute element is a similar size to the metal atoms, they will take the place of the metal atom in the grain structure and are called substitutional elements. Interstitial atoms are a smaller size than the atoms making up the metal matrix, which either allows the atoms to fill the extra space made by dislocations or impedes the movement of dislocations by causing strain in the lattice. Yu et al studied the effect of small amounts of oxygen (0.1%, 0.2%, and 0.3%) in hexagonally close packed alpha titanium [4]. They found that screw dislocations in titanium become pinned by interstitial interaction energy and a high barrier to the motion of oxygen atoms. This research also observed a non-linear relationship between increased yield strength and oxygen concentration (Figure 4). This experiment was done on samples on the nano scale, so the stress values found are higher than what is found on the macro scale. However, the strengthening mechanisms would be the same on a larger scale so the effect of oxygen content is still applicable.

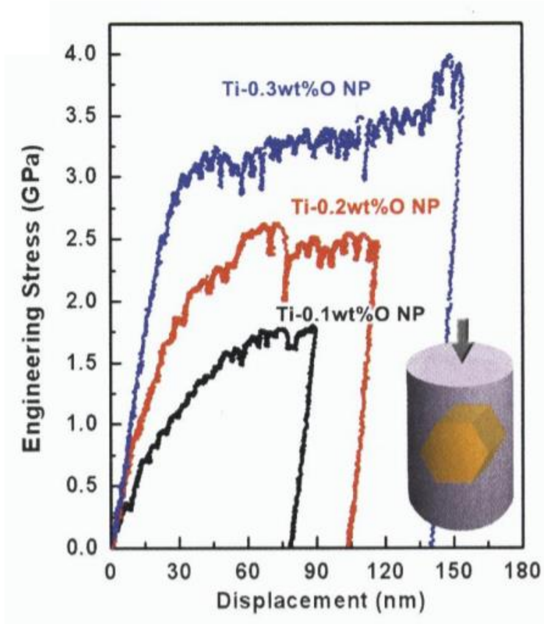


Figure 4: Compression test data for near alpha titanium [4]. Yield strength increases with increasing oxygen content.

Brandes et al also found that yield strength increases with increasing oxygen content when tensile and compressive tests were performed on unidirectionally rolled, commercially pure grade 1 and grade 4 titanium containing 0.036 wt.% and 0.330 wt.% oxygen (Table I) [5].

Table I. Test Data for Commercial Purity Titanium under Applied Tensile Stress *[5]*

Alloy	Oxygen Content (wt%)	E (GPa)	σys (MPa)
ASTM Grade 1	0.036	104.9	175
ASTM Grade 4	0.330	104.2	405

In industry, Ti-6-4 is classified into various grades depending on the amount of included interstitial content. Each of these grades has a target amount for each of the alloying elements, however, the allowable ranges for the different grades overlap. The compositional variation within a grade contributes to variation in the mechanical properties between material batches. In most cases the precision of the predicted mechanical properties is adequate for meeting the required specifications. The specific composition of a batch of material could potentially be used to predict the properties more precisely. For example, the expected strength could be compared between two batches of the same grade. Unfortunately, the numerical relationship between the interstitial content and the mechanical properties of forged Ti-6-4 have not been well quantified.

Two other interstitial elements that are often in titanium are carbon and nitrogen. These elements have the same strengthening effect as oxygen, but the magnitude of their effect varies. To make it easier to compare batches with different compositions, the amount of each of the three elements is sometimes combined into a single variable called oxygen equivalence. There is variation in the equations for oxygen equivalence because these equations are experimentally derived.

The oxygen equivalence equation is found by testing the material properties of many samples over a range of oxygen equivalence compositions. Then, a linear regression model is fit to the data. Coefficients are assigned to the weight percent of each element because they are known to influence mechanical properties by different amounts. Studies have been conducted on varying levels of all solute elements [6] and on near alpha titanium under compression [4].

An early study proposed that the equation for oxygen equivalence was Equation 2.

$$Oeq=O+2N+0.75C \qquad \text{Eq. 2}$$

.

Where oxygen (O), carbon (C), and nitrogen (N) are expressed in weight percent.

The studies used to calculate oxygen equivalence for titanium use a variety of titanium alloys and processing methods and examine different sets of resulting mechanical properties. Some of the differences in oxygen equivalence equations could be caused by these varying experimental conditions. A study from 2001 performed a regression analysis to determine the oxygen equivalence equation for titanium welds. A linear model was fit to each mechanical property individually. They found that the oxygen equivalence equation derived using just strength data, Equation 3, was slightly different than the equation found when trying to optimize several mechanical properties at the same time (Equation 4) [7]. In this study the authors included iron in the equation because it can also strengthen titanium. It is a substitutional element instead of an interstitial element so it is often not included in oxygen equivalence.

$$Oeq=O+2.69N+1.12C-0.21Fe \qquad \text{Eq. 3}$$

Where iron (Fe) is expressed in weight percent

$$Oeq=O+3.5N+2C-0.14Fe \qquad \text{Eq. 4}$$

Equations 3 and 4 show that the equations for oxygen equivalence may be different when optimizing strength versus toughness. However, this investigation focused on titanium welds and not forged parts. Therefore it is unknown if different oxygen equivalence equations should be used to predict each mechanical property for forged parts. The current equation used by Weber Metals is Equation 5.

$$Oeq = O+1.2N+0.67C \qquad \text{Eq. 5}$$

Fracture toughness describes the ability of a material containing a preexisting crack to resist crack propagation and fracture. This property is determined by composition and microstructure [6]. An inverse relationship between yield strength and fracture toughness has been well established [8]. Metal strength is dependent on resistance to plastic deformation, while high toughness requires ductility. Collins et al theorizes that the tradeoff between yield strength and fracture toughness is likely due to plastic deformation at the crack tip [6]. A higher yield strength will cause a smaller crack tip opening displacement. A decrease in the crack tip radius will result in high stress distribution in the nearby material.

1.3 Titanium Processing

Titanium is expensive to produce because it rarely exists in pure form. The material used in titanium ingots is typically a mixture of sponge extracted from titanium ore and recycled scrap metal. Any material discarded throughout the ingot and part making process is collected and recycled because of its monetary value. Impurities can be difficult to remove during processing, so certain amounts of contaminants must be expected [9]. However, suppliers and manufacturers are held to tight tolerances by their customers because alloy properties are heavily influenced by composition.

Vacuum arc remelt (VAR) is used to purify and refine standard titanium ingots (Figure 5) [10]. The titanium ingot, or electrode, is melted using a direct current arc (electrode negative, melt pool positive) in a vacuum between 0.1 to 1 Pa. As the electrode is heated and melts, the titanium drips down and solidifies into a new, homogeneous ingot. The VAR process can remove oxides, trace elements, and dissolved gases. The process is conducted under a vacuum so that the volatile contaminants can be removed. The “skin” layer around the final ingot contains the segregated nitrides and oxides and is alloy lean. The final skin layer must be removed before further processing is conducted. Titanium is typically melted twice using VAR to ensure homogeneity. To produce alloys, titanium sponge is blended with alloy containing material and then pressed into compacts to be used as electrodes in the first melting process.

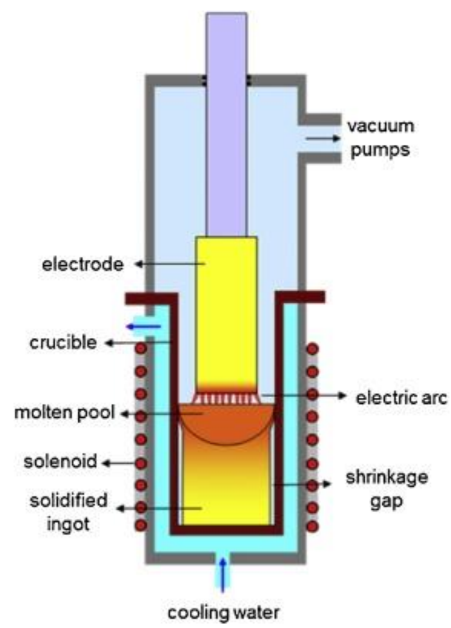


Figure 5: A VAR chamber contains the crucible, electrode, and electric elements. Water insulates the outside of the crucible.

Forging is used to obtain the rough shape of a component and form the desired microstructures. In this process, material is heated in a furnace to approximately 1750°F [9]. Open die forging is often done first to impart the desired amount of deformation and obtain the rough shape of the part. Closed die forging is then used to forge a shape much closer to the finished part. The resultant part can then be further machined and treated [11]. Titanium alloys are among the most difficult to forge because they require a higher applied stress to flow than many other metal alloys.

Forging titanium can cause the microstructure to be anisotropic and have different mechanical properties in different directions. For example, sheets of unidirectionally rolled titanium have significantly higher tensile strength in the transverse direction as compared to the longitudinal direction.

2 Experimental Procedure

In order to determine the strength and toughness of the different compositions of Ti-6Al-4V, tensile testing and fracture toughness testing was planned. Weber Metals chose and machined sections of previously forged parts into smaller pieces that could be more easily handled. The different sample compositions are denoted as compositions A-F in order of increasing oxygen equivalence. The sections of composition B were too large to handle as received, so each section was cut in half using a waterjet. Table II shows the approximate dimensions of each section. A two-part heat treatment was planned for the sections so that the microstructure could recrystallize post-forging. This process would also reduce microstructural variability between sections. The second annealing step relieved residual stress in the sections. The sections were then sent back to Weber Metals and machined into tensile and fracture toughness samples.

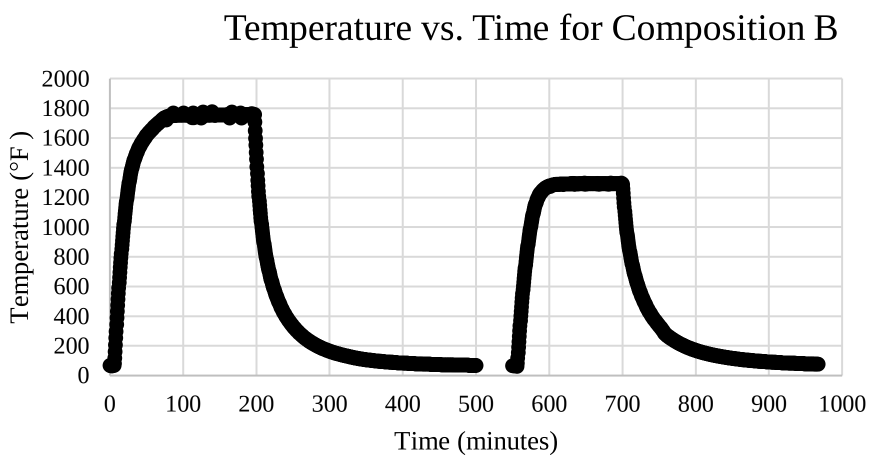
Table II. Approximate Section Dimensions

Composition	Number of sections	Length (in)	Width (in)	Thickness (in)
A	9	5.0	4.0	2.5
B	10*	6.0	3.0	2.5
C	1	6	4.2	2.3
D	1	5.6	2.4	3.9
E	1	4.4	3.8	2.5
F	1	5.9	3.5	2.0

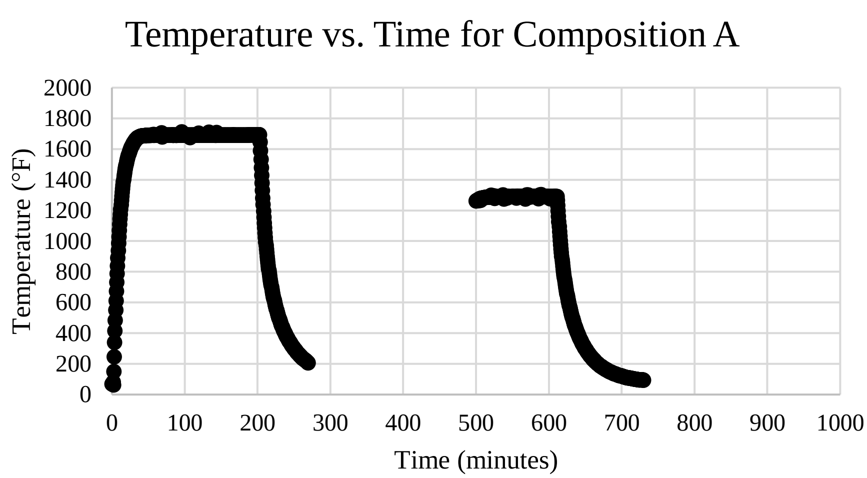
*After cutting in half

2.1 Heat Treatment

A two-part heat treatment was conducted on each section of titanium. Heat treatment was performed using two Fisher Scientific isotemp furnaces. One furnace was used to conduct the first heating step, while the other furnace was used for the second step. The furnace used for each step was kept consistent for each section to reduce the possible effect of temperature variation between furnaces. Due to the size limitations of the furnace, one section was placed in each furnace at a time. A large stainless-steel spatula was purchased to safely move the sections in and out of the furnace. A hole was drilled in one section of composition A and one section of composition B for a type K thermocouple to be placed during heat treatment. The thermocouple end was welded using equipment from the Mechanical Engineering department. Figure 6 shows the temperature profiles during heat treatment for both annealing steps. The temperature profile for composition A is slightly incomplete, but shows the same cooling rate.



(a)



(b)

Figure 6: Temperature profiles for (a) composition B, and (b) composition A, including both heat treatment steps. This profile confirms that the section reached the appropriate temperatures for the correct amount of time.

The profiles in Figure 6 are displayed in sequence for the sake of comparing the temperatures and times. Typically, the first heat treatment step was performed on a section one day, while the second step was performed the next day. This allowed simultaneous runs of the first and second heat treatment to be conducted on different sections.

The first step of heat treatment was a recrystallization anneal performed at 75°F below the calculated beta transus temperature (Equation 1). The purpose of staying below the beta transus temperature was to maintain the alpha + beta phases in the microstructure. However, a higher annealing temperature increases the rate of grain growth. The beta transus temperature for each composition is included in Table III. The annealing process consisted of one hour for the section to reach the correct temperature followed by two hours held at the annealing temperature. The section was subsequently removed from the furnace and allowed to air cool (Figure 7).

The rate of cooling influences alpha particle growth in the microstructure while above 1300°F. In this case, the initial cooling rate was determined via thermocouple to be 90°F per minute. Weber Metals uses fans to force air over their parts after the recrystallization anneal to cool the parts more rapidly. The parts that they produce are significantly larger than the pieces we heat treated and would cool slower under the same cooling conditions. Therefore, cooling small sections using natural convection should result in a similar cooling rate compared to cooling full sized parts using forced convection.

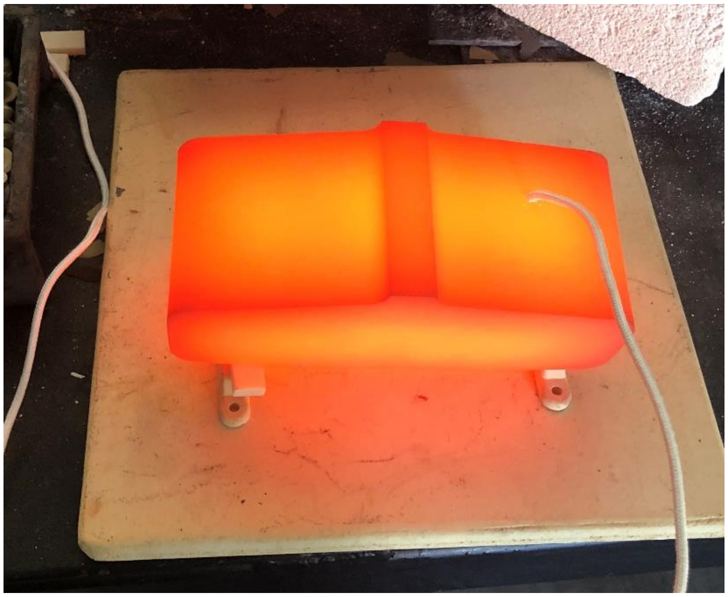


Figure 7: The thermocouple was placed in the drilled hole of a section of composition B.

The second mill anneal was performed at 1300°F and consisted of one hour for the section to reach the correct temperature and one hour held at that temperature. The section was then removed and allowed to air cool again.

Each section had a thin layer of brittle oxide on the exterior after heat treatment, known as an alpha case. The alpha case was easily removed in some cases. Any remaining alpha case would be removed by subsequent sample machining.

Table III. Beta Transus Temperature for each Composition

Composition	Al (wt%)	O (wt%)	N (wt%)	C (wt%)	V (wt%)	Fe (wt%)	Beta transus (°F)
A	6.00	0.20	0.003	0.007	4.04	0.17	1805
B	6.43	0.18	0.01	0.020	4.19	0.22	1842
C	6.62	0.19	0.018	0.026	4.26	0.22	1866
D	6.32	0.18	0.014	0.033	4.16	0.22	1856
E	6.38	0.20	0.016	0.031	4.12	0.23	1864
F	6.15	0.19	0.017	0.036	4.16	0.22	1857

2.2 Tensile and Fracture Toughness Testing

After heat treatment was completed, the sections of material were returned to Weber Metals. Weber Metals sent them to Atlas Testing Laboratories, where they were machined into tensile and fracture toughness samples using ASTM standards E8 and E399 accordingly [12] [13]. The number of samples machined in each direction is displayed in Table IV.

Table IV. Sample Type Distribution

	Tensile		Fracture toughness	
Composition	Longitudinal	Transverse	L-T	T-L
A	5	5	4	0
B	10	10	5	5
C	1	0	1	0
D	1	0	1	0
E	1	0	1	0
F	1	0	1	0

Tensile samples were machined in the longitudinal and transverse directions relative to the forging flow directions. Fracture toughness samples were machined in the L-T and T-L directions (Figures 8-9). Having samples machined along the major directions increases the scope of inference for potential conclusions. If the directions were not considered, and the samples were machined in random directions, there would be more variation in the resulting data. If only one direction were used it would limit the conclusions to that specific direction. However, similar tensile strength in both directions is expected.

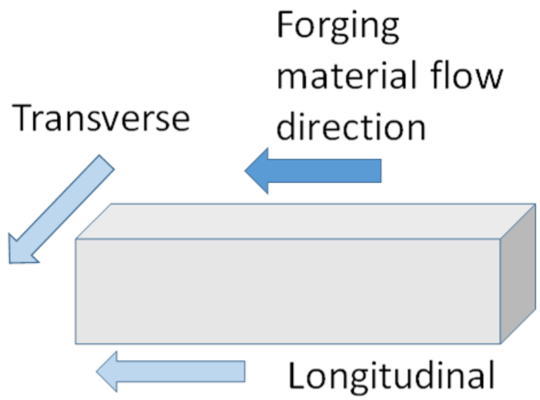


Figure 8: Diagram of forging directions. Tensile samples were machined along both major directions.

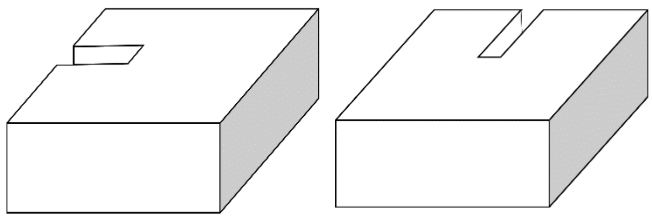


Figure 9: Diagram of fracture toughness samples in relation to the directions in Figure 8. Left is the L-T direction, while right is the T-L direction. The starting notch is in two different orientations.

Tensile tests of samples of composition A and composition B were conducted using an Instron 5584 and proper sample fixtures. Tensile tests of compositions C-F were conducted by Weber Metals. Fracture toughness testing was conducted by Atlas Testing Laboratories.

2.3 Metallography

Pieces of composition B were sectioned using an abrasive saw in both the transverse and longitudinal directions. The samples were mounted in bakelite and polished. The samples were etched using Kroll's Reagent (1.7% hydrofluoric acid, 6% nitric acid) for approximately five seconds and then examined under a microscope.

3 Results

The oxygen equivalence was calculated for each composition in order to compare the results of mechanical testing (Table V). Oxygen equivalence values were calculated using the composition reported by the titanium supplier.

Table V. Oxygen Equivalence for each Composition

Composition	O (wt%)	N (wt%)	C (wt%)	Oeq
A	0.20	0.003	0.007	0.1623
B	0.18	0.010	0.020	0.2004
C	0.28	0.014	0.033	0.2189
D	0.19	0.018	0.026	0.2290
E	0.29	0.017	0.036	0.2345
F	0.20	0.016	0.031	0.2400

3.1 Strength and Toughness Data

The tensile strength data collected is displayed in Table VI. The sections of compositions C-F were only large enough to machine one tensile sample each.

Table VI. Tensile Strength Data

Composition	Longitudinal (ksi)	Transverse (ksi)
A	116.8	128.3
A	128.6	120.4
A	120.7	121.0
A	132.4	128.2
B	141.1	141.2
B	139.5	137.6
B	141.0	139.1
B	140.3	140.8
B	138.9	138.3
B	138.9	140.3
B	139.1	137.9
B	140.1	126.0
B	139.6	134.2
B	144.6	132.3
C	141.0	
D	147.9	
E	142.7	
F	143.9	

The standard deviation for the tensile strength was relatively high. There was a statistically significant increase in strength with increasing oxygen equivalence ($p< 0.001$). This means that the average tensile strength is different between the populations. However, the ranges for strength overlapped between compositions (Figures 10-11). One of the assumptions of a linear regression model is equal variance. The amount of variation in the strength of composition B is higher than expected and higher than that of batch A. For the higher interstitial batches there was only one data point per composition so the variation could not be calculated for those compositions. Due to these two factors, we determined that it would invalid to assume equal variance so a linear regression model should not be performed.

There was no significant difference in strength between the transverse and longitudinal directions.

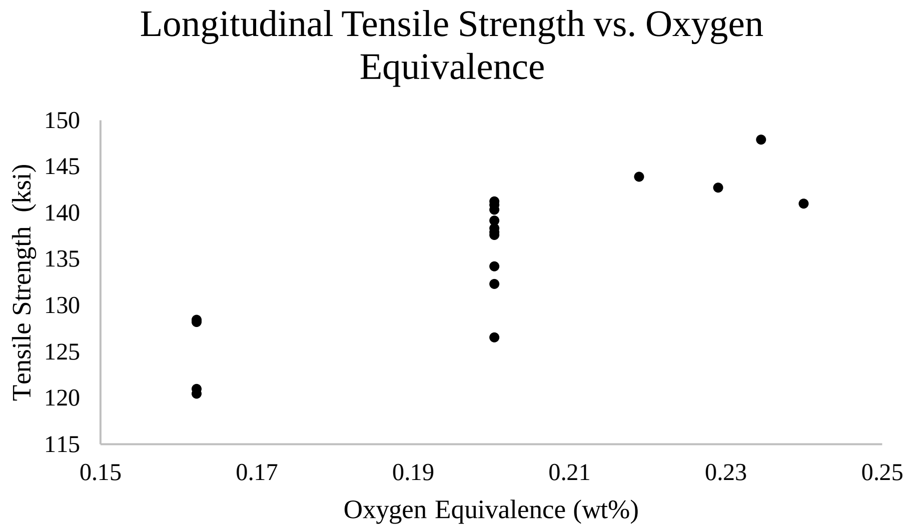


Figure 10: Tensile strength in the longitudinal direction is plotted against oxygen equivalence. There is a general increasing trend.

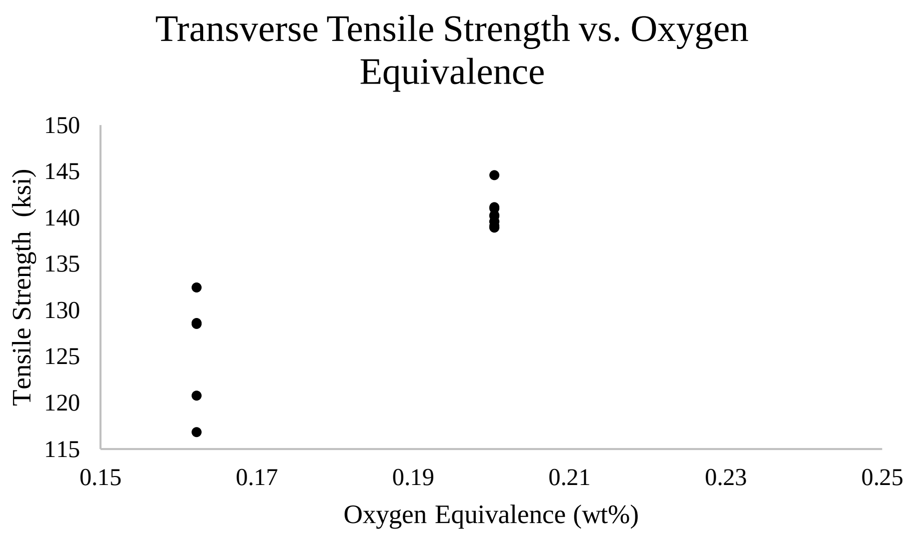


Figure 11: Tensile strength in the transverse direction is plotted against oxygen equivalence. There is a general increasing trend.

The fracture toughness testing was only able to determine the conditional toughness value, K_Q, rather than the linear-elastic plane-strain fracture toughness of the samples (K_{IC}) because the samples did not meet the geometry requirements specified in ASTM standard E99-20. However,

K_Q and K_{IC} are often considered to be comparable. Fracture toughness data is reported in Table VII.

Table VII: Fracture Toughness Data

Composition	L-T Direction (ksi*in^0.5)
A	78
A	75
A	78
A	76
B	79
B	80
B	80
B	79
B	77
C	75
D	70
E	75
F	63
Composition	T-L Direction (ksi*in^0.5)
B	68
B	63
B	73
B	69
B	65

L-T directional toughness was compared to oxygen equivalence because the set of T-L data was incomplete (Figure 12). A linear model was fit to the data; however, it did not fit well (R-squared = 0.29). The lack of fit test was significant ($p = 0.0003$). This means that the error in the model is more due to the model not fitting the data well as opposed to variation between samples at the same condition. These two factors indicate that a linear model should not be used for this data.

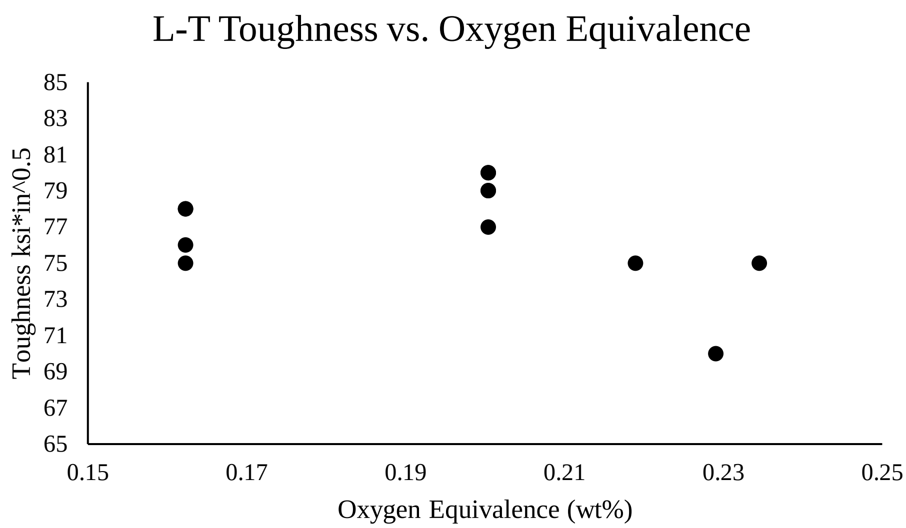


Figure 12: Toughness and oxygen equivalence are plotted together. There is no significant trend in the L-T direction.

T-L data is only available for composition B because most of the samples were not large enough to machine T-L testing samples. Therefore, we are unable to include sample direction in the model for toughness vs. oxygen equivalence. A two-sample t-test with unequal variance was conducted to test the difference of toughness with direction. The L-T samples were statistically significantly higher than the T-L samples with a p- value of 0.0008 and an average difference of 11.4 ksi*in^0.5.

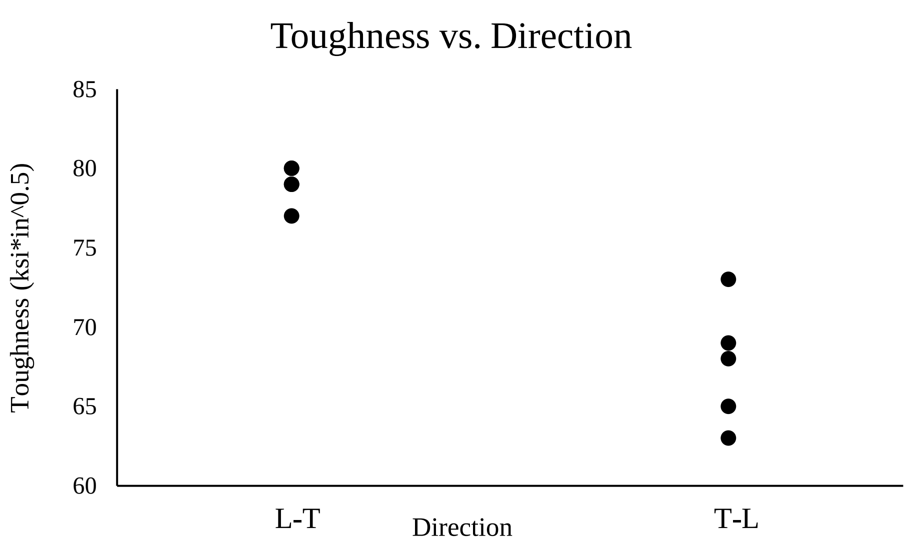


Figure 13: Toughness values are separated by sample direction. There is a significant difference between the two directions.

There is no clear trend present when tensile strength and toughness are plotted together (Figure 14). The sample compositions are separated by color.

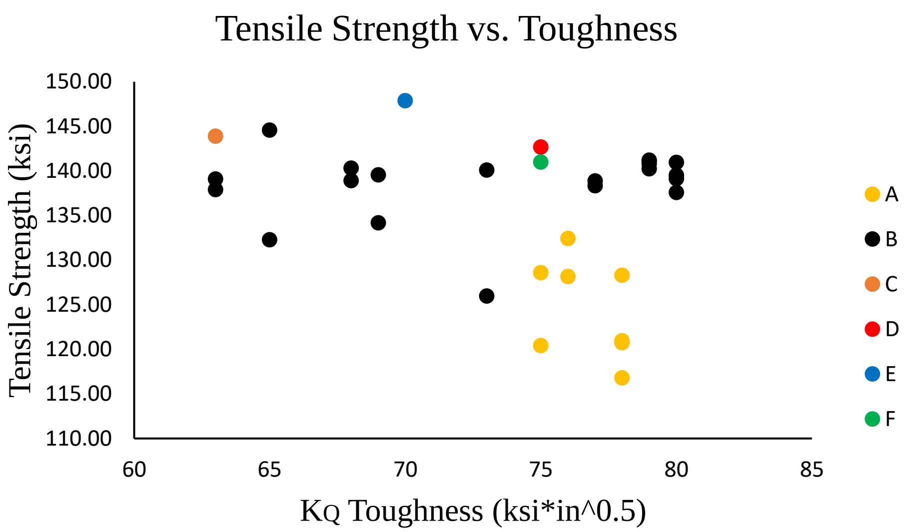


Figure 14: Tensile strength and toughness are plotted together for comparison. The data includes all sample directions.

3.2 Microstructure

The resulting micrographs show equiaxed alpha phase (light regions) in a transformed beta phase matrix (dark regions). Forging and subsequent annealing caused the alpha particles to grow. (Figure 15). Similar tensile strength in the longitudinal and transverse directions can be attributed to the equiaxed microstructure.

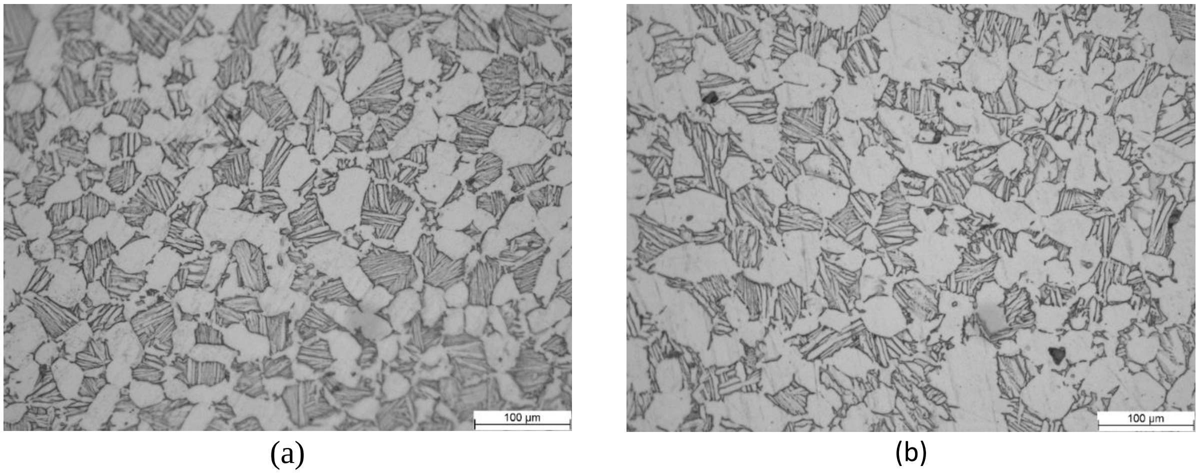


Figure 15: Micrographs were taken (a) in the longitudinal forging direction and (b) the transverse direction (200x).

4 Discussion

A general trend is apparent in the oxygen equivalence versus tensile strength data; however, a linear model could not be fit. Tensile strength data for compositions A and B displays a high variation. Because only one tensile sample was tested from compositions C-F, the potential variation in the tensile strength of C-F is unknown.

Even if there had been more samples for compositions C-F, the high amount of variance would make the model impractical for use. Interstitial content is being investigated to predict the properties of a component more precisely than by just using the grade of the metal. With such a high variance to the model, there would be a wide confidence interval for any prediction for tensile strength. Therefore, the prediction using oxygen equivalence would likely not be more precise than that for the grade overall.

Although the testing direction was not found to be significant for tensile strength, it was still valuable to include it in the experiment. If only one direction were used, the scope of inference for the model would have been limited to the direction tested. A similar strength in the longitudinal and transverse directions was expected based on past results from Weber Metals.

The fracture toughness data did not have a linear correlation with oxygen equivalence as expected from the literature. It is unclear if the relationship was not linear due to errors in the data from this experiment or if a linear model is inappropriate for the actual relationship between oxygen equivalence and toughness. Further testing could be done to determine which of these is the case.

The plot of tensile strength vs toughness (Figure 14) does not show any clear trend between the two properties. Literature suggests that the two properties are expected to be inversely related. This lack of trend could be due to the small number of samples.

The large amount of variation in the data could be caused by a number of factors. The oxygen equivalence values were calculated based on chemical compositions reported by the titanium supplier. The standard practice for testing the composition is removing material from the top and bottom of the ingots. Alloy segregation during ingot purification can cause a non-uniform compositional gradient throughout the boule. Therefore, there is a small amount of variability in the oxygen equivalence.

5 Conclusions

1. The effect of increased oxygen equivalence on the tensile strength of Ti-6Al-4V cannot be found numerically due to lack of fit in the data.
2. The effect of increased oxygen equivalence on the K_Q toughness was not found to be statistically significant.
3. Sample direction was found to be statistically significant for the toughness of composition B.

4. Sample direction was not found to be statistically significant for the tensile strength of compositions A and B.

6 Recommendations

1. The data on tensile strength and fracture toughness found in this study cannot be used to predict the properties of similarly treated parts.
2. Testing a larger number of samples with more distinct compositions is required to find the numerical correlation between oxygen equivalence and strength and toughness in Ti-6Al-4V.

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