Investigation into Oxygen Diffusion as it Relates to Alpha Case Formation in Ti-6Al-4V Based on Different Forging Thermal Cycles

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By

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Abstract

An investigation into the growth kinetics of alpha case in Ti-6Al-4V in the presence of oxygen was conducted for different forging thermal cycles. This was done to determine whether the alpha case should be removed after each thermal cycle or as the final step after forging. The thermal cycles consisted of four hours at 1750 °F. Samples were subjected to this treatment one, two, three, and four times along with a continuous sixteen hour hold. Four cycle and Hold samples were subjected to an additional thermal cycle above the beta transus at 1925 °F. These then went through a final stress relieve cycle at 1350 °F for four hours. The alpha case depth was measured for each of these samples using three methods. These include area fraction analysis using ImageJ, visual estimations, and Vickers microhardness testing. Each of these three methods showed a large initial case growth followed by diminishing growth rates. Area fraction was the most conservative measurement resulting in significantly higher values for depth than the other two methods. Visual estimations and Vickers hardness corroborated each other’s values. After one four hour thermal cycle at 1750 °F the case depth by visual estimation is 250 micrometers. The second, third, and fourth thermal cycles are 366, 388, 392 micrometers respectively. After four thermal cycles at 1750 °F, samples were heated to 1925 °F for half an hour resulting in a decrease in case depth to 292 micrometers, and a subsequent heat treatment at 1350 °F for four hours resulting in a depth of 263 micrometers. The sixteen hour hold samples undergo the same 1925 °F and 1350 °F cycles with similar results. The Hold has a depth of 537 micrometers, which first decreased to 318 and then to 281 micrometers.

Keywords

Metallography, Materials Engineering, Ti-6Al-4V, Forging, Alpha Case, Titanium Metallurgy, Microstructure, Oxygen Diffusion, Vickers Hardness, Beta Transus
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1. Introduction

Titanium is an indispensable material for aerospace and high-performance applications due to its high strength to weight ratio compared to steel or aluminum. Pure titanium is an allotropic material, meaning it has multiple stable phases. These phases are alpha, a hexagonal closed pack (HCP) structure at room temperature, and beta, a body centered cubic (BCC) phase above 1925 °F. The most common titanium alloy, Ti-6Al-4V, is an alpha-beta alloy. This means both the alpha and beta phases are present at room temperature [1]. Different elements work to stabilize each of these phases. Oxygen is a key example of an alpha stabilizer. This coupled with high amounts of surface diffusion leads to the formation of alpha case, a hard and brittle layer on the surface of heated titanium components. This layer must be removed before a forged piece is considered finished. This is usually done with chemical milling to dissolve the alpha case. In order to accurately know how much material to remove, a comprehensive understanding of alpha case growth and depth is required.

Titanium’s high strength to weight ratio makes it a desirable metal to use for high performance applications. Understanding its allotropic nature is essential to using it to its maximum potential. Experimentally determined growth rates of alpha case during specific forging conditions are important to know so that it can be properly addressed to lessen tool wear, minimize imperfections and lead to the highest quality forgings possible. Alpha case growth increases in depth with increased time and temperature. This project will address the extent of alpha case growth on Ti-6Al-4V heated to 1750 °F for 4 hours for one, two, three, and four times, along with being held for a continuous 16 hours. After completing either four thermal cycles or 16 continuous hours, samples are then heated above the beta transus at 1925 °F for 30 minutes then examined. An additional set of samples that underwent the previous two processes will be heated at 1350° F for 4 hours and examined. These parameters were chosen to closely match the worst case forging conditions that titanium will experience at Weber Metals.

1.1 Overview of Titanium Metallurgy

Different alloying elements can stabilize either the alpha or beta phases. For example, aluminum, oxygen, nitrogen, and gallium are alpha stabilizers while molybdenum, vanadium, tungsten, and tantalum are beta stabilizers. In general, alpha alloys are stronger but have lower ductility than beta alloys. The higher ductility of the beta alloys is due to the greater amount of slip planes in the BCC Structure [1] [2]. Titanium has four major categories, alpha alloys, near alpha alloys, beta alloys, and alpha-beta alloys. These categories describe the general microstructures of titanium by identifying the stable phases present after it has been processed [3]. It is important to note that when discussing titanium, microstructure and crystal structure are not synonymous terms. Both the grain structure, sometimes called microstructure,
and the crystal structure of each grain contribute to the overall properties of titanium [3]. Alloying, which effects the crystal structure, and the processing, which effects the microstructure, are crucial to understanding the properties of a titanium component [1].

### 1.1.1 Alloying Elements for Titanium

Alloying elements for titanium are either alpha stabilizers or beta stabilizers. The function of these stabilizers is to widen the temperature ranges at which each phase is stable. One important component of this alpha to beta phase transformation is a temperature known as the beta transus temperature. The beta transus is the temperature at which alpha or alpha plus beta alloys will transform entirely to the beta phase [3]. Alpha stabilizers raise the beta transus temperature and cause less beta to be retained. Common alpha stabilizers include aluminum, tin, zirconium, oxygen, and nitrogen [3]. Nearly all titanium alloys include one or more of the metal alpha stabilizers because they improve the creep strength in the alpha phase [3]. In Ti-6-4 aluminum is the alpha stabilizer.

Beta stabilizers lower the beta transus temperature and cause more beta titanium to be retained [3]. Beta stabilizers fall into two major categories that are distinguished by the mechanisms they use to lower the beta transus temperature. Ti-6-4 uses vanadium as its beta stabilizer. The first group is the beta isomorphous group, which involves the alloying elements being miscible in beta titanium [3]. This group includes elements such as molybdenum, vanadium, and tantalum [3]. The eutectoid group is the other group of beta stabilizers and includes elements such as manganese, iron, and chromium. The elements in this group have a much higher solubility in beta titanium than in alpha titanium [3].

### 1.1.2 Titanium-6Al-4V and Other Alpha-Beta Alloys

Alpha titanium has a hexagonal close packed crystal (HCP) structure (Figure 1). While it has good creep and corrosion resistance, alpha alloys are typically difficult to age harden, but, as a result, are easy to weld [3] [1]. One of the most encountered alpha stabilizers is oxygen, which alloys interstitially. These interstitial oxygenes place strain on the lattice causing an increase in strength and decrease in ductility [1].

*Figure 1: HCP crystal structure for alpha titanium [1].*
Compared to alpha titanium, beta titanium has a body centered cubic crystal structure and a chemical composition that causes it to retain the beta titanium structure through cooling (Figure 2) [3]. BCC titanium has three octahedral interstitial site per atom while HCP only has one octahedral interstitial per atom. This one octahedral site is larger, allowing the solubility of oxygen, nitrogen, and carbon to be much higher in the alpha phase [4].

![Figure 2: BCC crystal structure and close packed plane of beta titanium [1].](image)

Alpha-beta alloys have grains of both HCP and BCC crystal structures. This alloy composition that permits a complete transition to beta titanium upon heating, but upon cooling transforms back to alpha with some retained beta titanium [3]. Alpha-beta alloys are critically important for a variety of applications and are the main type of titanium used. Alpha-beta alloys comprise roughly seventy percent of the entire United States’ titanium market [2]. Fifty percent of the entire world market is dominated by a single alpha-beta alloy: Ti-6Al-4V [1]. Ti-6Al-4V tops the market because of its well-rounded balance of mechanical properties. This alloy has been the subject of extensive research and development, making it one of the best understood titanium alloys [1].

### 1.1.3 Formation of Alpha Case

Beta titanium can transform into alpha titanium through multiple methods such as a martensitic transformation or diffusion. The method of transformation is determined by the processing and alloy chemistry of the component [3]. When beta titanium transforms into alpha there is a tendency for it to form an acicular, scaled, or plate like structure. When Ti-6Al-4V is slow cooled from above the beta transus temperature it forms these acicular plates with a specific crystallographic relationship to its parent beta phase (Figure 3) [3]. The new HCP alpha phase will have its basal plane form parallel to the close packed plane of the beta phase [3].
It should be noted that the formation of alpha titanium often occurs most prominently on the outside of components [5]. This is because oxygen is an alpha stabilizer so the outer layer of components will absorb oxygen which acts as an alpha stabilizer [5]. This oxygen rich outer layer is alpha case. This heightened presence of oxygen is demonstrated by Figure 4. This is also a large contributor to its high price as it must be held under an inert atmosphere or vacuum during the melting process. Titanium’s reactivity with oxygen also provides titanium with its chemical resistance due to the formation of a stable oxide layer. This oxide layer also acts as the site where oxygen atoms diffuse into the titanium component. The diffusion of oxygen from the surface results in a continuous layer of oxygen stabilized alpha titanium often called an alpha case. The low ductility of this alpha layer makes it susceptible to crack formation when loaded under tension [1].
The formation of this alpha case is only of concern at temperatures above 550 °C as below that the oxygen diffusion is insignificant [1]. Different alloying elements can be added to titanium to decrease the diffusion of oxygen into titanium and thus decrease the formation of the alpha case. These elements are aluminum, silicon, chromium, niobium, tantalum, tungsten, and molybdenum. Aluminum is considered the most effective method of reducing oxidation because it is reactive with oxygen and will form a mixed layer of titanium oxide (TiO₂) and alumina (Al₂O₃) just below the initial titanium oxide layer [1]. The maximum temperature for titanium applications is roughly 600 °C.

1.2. Diffusion

Diffusion is a type of mass transfer undergone by solids and involves both the movement and transport of atoms [6, 7]. There are a variety of subcategories and mechanisms by which diffusion can occur. Most important to titanium metallurgy and forging is a type of free diffusion called interdiffusion. Free diffusion means that there are no external mechanical or electronic forces driving it, just a difference in concentration between the parts of the system. Interdiffusion relates to the parts of the system. It means that the diffusing species and the host species are different. In this case, oxygen diffuses into titanium. The two mechanisms by which this can occur are vacancy and interstitial diffusion [6, 7, 8]. These mechanisms determine how the diffusing species moves through the host species’ lattice. Both are referred to as lattice or volume diffusion [7]. Vacancy diffusion occurs through unoccupied lattice sites. The diffusing species uses the unoccupied lattice sites to move about its crystalline host. However, this is less common in metal alloys and more common during self-diffusion of pure metals [6]. Interstitial diffusion is significantly more common due to the diffusing atoms decreased size and increased mobility [6, 7, 9]. The interstitial diffusion mechanism entails the diffusing species, which is usually much smaller than the host atoms, travelling through the interstitial sites [6, 7, 8].

1.2.1 Factors Affecting Diffusion

There are several factors that affect the rate at which diffusion occurs. The main factor affecting diffusion is the aforementioned difference in concentration. More commonly, it is referred to as the concentration gradient because of how it is represented in Fick’s 1st Law that deals with steady state diffusion (Eqn. 1).

\[ J = -D(\frac{\partial c}{\partial x}) \]  

The concentration gradient is the \( \frac{\partial c}{\partial x} \) term. The flux of atoms (J) expresses the mass of atoms moving through an area for a given time. The D represents a value called the diffusion coefficient. This value
relates the ability of a species to diffuse to key factors that affect diffusion. The diffusion coefficient is calculated using an Arrhenius equation with a standard diffusion coefficient $D_0$ shown in (Eqn. 2).

$$D = D_0 e^{-Q/kT} \quad (2)$$

The exponential term contains activation energy divided by Boltzmann’s constant and temperature. Activation energy and the standard diffusion coefficient are material specific values and are unaffected by factors like the concentration gradient and temperature. Temperature is the only value involved that actively affects diffusion. Both a greater difference in concentration in Fick’s 1st Law and greater temperatures in the Arrhenius equation increase the rate of diffusion.

### 1.2.2 Diffusivity of Titanium

The standard diffusivity in a solid is empirically determined as well as the activation energy for that diffusion. Diffusivity values are unique to both the host material and the species diffusing into it. Alpha and beta titanium have different crystal structures with varied sizes and placement of their interstitial sites. This means that they have different rates of diffusion that contribute to that of Ti-6Al-4V, an alpha-beta titanium alloy. The standard diffusivity value for Ti-6Al-4V is $1.1 \times 10^{-15}$ m$^2$/s with an activation energy of 156 kJ/mol [10, 11]. This means it has an atomic packing factor (APF) of 0.74, higher than that of beta titanium [8]. Despite this, alpha titanium has a lower activation energy, but does demonstrate a lower diffusivity than beta titanium [12]. While titanium is known to form a stable oxide that increases chemical resistance, the oxide layer also acts a diffusion interface [1]. Oxygen from the surface diffuses further into the interior and results in a continuous layer of oxygen stabilized alpha titanium often called an alpha case [1]. Between the relevant forging ranges of 932 °C and 1142 °C the diffusivity of oxygen in alpha titanium can be represented by (Eqn. 3).

$$D_\alpha = 0.778 \frac{\text{cm}^2}{\text{s}} e^{(-48600 \text{ J/mol})/RT} \quad (3)$$

The beta titanium crystal structure has an atomic packing factor of 0.68 [8]. Because this structure is more spacious, it is able to diffuse oxygen through its interstitial sites more easily. The activation energy, however, remains higher than that of alpha titanium. Between the temperature range of 932 °C and 1142°C the diffusivity of oxygen in beta titanium can be represented by (Eqn. 4).

$$D_\alpha = 3.30 \times 10^2 \frac{\text{cm}^2}{\text{s}} e^{(-58800 \text{ J/mol})/RT} \quad (4)$$
1.3 Titanium Forging and Processing

Forging is the process of hot working a metal into a desired shape. The basic principles of forging have changed little in the scope of human history. Ultimately, the process involves a piece of metal being heated and plastically deformed. While the process has remained the same, the tools of the trade have changed significantly. Modern forging plants often use large hydraulic presses and closed dies to produce high-precision parts (Figure 6). The alloy composition determines most of the physical properties such as density or thermal expansion, but the processing of a titanium component will allow for selective balancing of a component’s properties [1]. This balancing of properties occurs because of changes in a component’s microstructure.

1.3.1 General Steps of the Forging Process

The first step in forging is to heat the metal, usually in a large furnace. At Weber Metals, a typical Ti-6Al-4V forging is heated to 1750°F. Close die forged components are often forged in multiple steps depending
on the complexity of the model. The first step is called potting or roughing which gets the part either close to the desired shape or to a similar aspect ratio of the final part. Some forging steps for different parts can be done in parallel. Between each pressing the piece is reheated. In alpha-beta alloys like Ti-6Al-4V this heating typically remains below transus temperature, but forging can also be done above the beta transus to lower residual stress and increase the ductility of the part [1]. This is typically done when damage tolerance is a priority.

Once components are forged to their desired shape, heat treatment can be done to remove residual stress or affect grain sizes. In the case of Ti-6Al-4V at Weber Metals, a brief heat treatment above the beta transus (1925°F) is done to improve ductility. This is followed by a stress relief step at 1350°F for two to four hours to relieve some residual stress. Figure 7 shows the entire forging and heat treatment process.

![Figure 6: Potential heat treatment plan of Ti-6Al-4V at Weber Metals with a blue line representing the beta transus temperature.](image)

Once the heat treatments are completed, the components are subjected to surface treatments. These surface treatments can include sand blasting, shot peening, chemical milling, or some combination of treatments. The purpose of these is to remove any lubricant coatings from the forging process, impart surface stress to increase strength, or remove alpha case, respectively [13]. Components can then be machined into final, precise dimensions.
1.3.2 Alpha Case Formation and Mitigation During Forging

There are several known techniques that can assist in the mitigation of alpha case formation during the forging process. Alpha case growth is a temperature and time dependent process [7]. By limiting the amount of time titanium spends in the furnace and during forging alpha case can be minimized. Carefully choosing the furnace temperature to only heat the titanium as high as necessary also limits the formation of alpha case growth. The other methods rely on the fact that diffusion operates based on concentration gradients. If the heating and forging is conducted in an inert atmosphere, only the surface oxygen will be available to diffuse into the titanium. This method can also be implemented by using a coating instead of a controlled atmosphere. This is often done in the form of a glass coating that both protects the parts surface from the atmosphere and lubricates the part during forging [14]. Alpha case greatly increases the hardness and brittleness of the surface of a forged titanium component. This brittle layer is easier for cracks to nucleate in compared to the bulk material.

1.3.3 Removal of Alpha Case

Chemical milling (chem milling) is the method most often used to remove alpha case from the surface of forged titanium components [15]. Chem milling is the process of submerging the component in a temperature regulated acid bath to dissolve the surface layer. This bath consists of 6-16% hydrofluoric acid (HF) and about 10% nitric acid (HNO₃), with water to balance. [15]. The experiment run by Vinod et. al. showed an alpha case growth of 63 μm in Ti-6Al-4V after 1 hour at 930°C. This alpha case was completely removed by a solution of 8-9% HF and 10% HNO₃ after 15 minutes. The remaining case depth for various chem milling solutions is shown in Figure 8.

![Figure 7: SEM images of remaining alpha case depth after fifteen minutes of chemical milling in various etchant mixture combinations [15].](image)
The HF dissolves the oxide scale and the titanium itself. The HNO$_3$ works as an oxidizing agent and repassivates the surface [7]. This process is exothermic so continuous cooling of the chem mill bath is required.

2. Experimental Procedure

The experiment is designed to replicate the forging process at Weber Metals. The process starts with samples of Ti-6Al-4V cut from a test ring to approximately ¾" x ¾" x 1 ¼". The samples were subjected to various thermal cycles. Two replicants were used for each treatment. The first treatment was four hours at 1750°F. Samples were subject to this treatment 1, 2, 3, and 4 times. These are referred to as 1, 2, 3, and 4, with the replicants being labeled A and B. Table I is the breakdown of the naming convention. These samples were used to determine the growth kinetics of alpha case after multiple thermal cycles. A sixteen-hour hold at 1750°F was conducted and compared to samples 4. Samples subjected to the 4 and H treatment were then subjected to a 30-minute heating at 1925°F to improve ductility. This is above the beta transus temperature of titanium. 4β and Hβ were then subjected to an additional treatment at 1350°F for four hours to relieve stress.

Table I: Thermal Cycles Compared to Names

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>1750 °F</td>
<td>1A, 1B</td>
</tr>
<tr>
<td>4 hours</td>
<td>2A, 2B</td>
</tr>
<tr>
<td>4 hours</td>
<td>3A, 3B</td>
</tr>
<tr>
<td>4 hours</td>
<td>4A, 4B</td>
</tr>
<tr>
<td>16 hours</td>
<td>HA, HB</td>
</tr>
<tr>
<td>4 hours</td>
<td>4βA, 4βB</td>
</tr>
<tr>
<td>16 hours</td>
<td>HβA, HβB</td>
</tr>
<tr>
<td>4 hours</td>
<td>4βTA, 4βTB</td>
</tr>
<tr>
<td>16 hours</td>
<td>HβTA, HβTB</td>
</tr>
</tbody>
</table>

2.1 Thermal Cycles

Twelve samples of titanium were loaded into a Fisher Isotemp muffle furnace at 1750°F for the first four hour cycle. A type K thermocouple was used to establish the furnace’s hot zone and that it had truly reached 1750°F. Samples were placed on a ceramic tray which could be moved in and out of the furnace with tongs (Figure 9). Samples were allowed to fully cool before being reloaded in the furnace. Ten
samples underwent a second cycle, eight underwent a third cycle, and six underwent a fourth cycle. Six unheated samples experienced 1750 °F for sixteen hours.

![Figure 8: Samples were placed in the furnace such that no samples were in contact with one another on a ceramic tray.](image)

A type K thermocouple was also used to verify the temperature of the furnace could reach 1925°F as this was nearing the furnace’s maximum temperature. Four samples from the four cycle and four from the sixteen hour thermal cycle were placed on the tray with care to not mix the two different thermal cycles. They were loaded into a 1925°F furnace for thirty minutes. Two samples from each thermal cycle that underwent 1925°F temperatures were finally heated to 1350°F for four hours.

### 2.2 Metallography and Image Analysis

Once the thermal cycles were complete, samples were then cut in half with a rubber bonded silicon carbide blade to reveal their cross sections. These were then mounted in Bakelite. Polishing steps went from 240, 320, 400, 600, 800, 1200 grit emery paper to 6 micron, 3 micron, 1 micron, to 0.5 micron polishing wheels. Polished samples were etched in Kroll’s Reagent, a solution consisting of 5% nitric acid and 2% hydrofluoric acid, for 15 seconds. The etched samples were rinsed with water and then isopropyl alcohol. Three micrographs of each sample were taken at different regions with care to avoid the bottom edge and any corners. The two replicants and three images of each sample provided six data points for each treatment to average into the case depth. The alpha case depth was measured using three methods: area fraction, visual estimation, and microhardness traversal.
2.2.1 Area Fraction Analysis
Area fraction analysis was done by importing the micrographs into ImageJ, an open source software package that is used for taking calibrated measurements of images. The micrographs were made binary so that the software could recognize what was alpha and what was beta (Figure 10). Percent alpha phase by area was calculated in the core while the measurement was moved towards the edge of the image. When the percent of alpha increased, that was the furthest edge of total alpha case. This distance to the edge of the sample is the case depth using this method. Area fraction analysis was the most conservative of all measurements and registered the smallest increase in percentage of alpha grains.

![Figure 9: Micrograph of 2A at 100x magnification with scale bar equal to 100 µm (a) as taken and (b) made binary in ImageJ.](image)

2.2.2 Visual Estimation
Visual estimation by an expert eye is considered a valid method to evaluate case depth in industry. This method was used because the area fraction method was shown to be too conservative. Visual estimation was done by randomizing the order of the micrographs, then importing them into ImageJ. Each of the three team members determined the point they believed the alpha case ended on an as taken micrograph. In this case, ImageJ was used only as a measuring tool rather than an analysis tool. These values were averaged to determine the case depth for each sample.

2.2.3 Microhardness
The third method was microhardness traversal. In this method hardness measurements were taken using a Buehler Micromet II Microhardness Tester with a Vickers indenter at a 500 gram load. Hardness measurements were taken starting 100-125 microns from the edge of the sample. Following the ASTM E384 standard, the indents had to be 2.5 times the distance of the diamond-shaped indents apart [16]. This was done to evaluate the alpha case thickness by a criterion that is directly related to the material properties of the sample. Evaluating the case thickness by hardness should yield a case depth that is less
conservative and more accurate than that of area fraction analysis. To accomplish this, we move the indenter in towards the core 25 microns and down along the edge 150 microns between each measurement (Figure 11). If the points are taken too closely together then stress field interactions can lead to erroneously high hardness values. These measurements were taken until the hardness leveled off to a value consistent with that of the core. Alpha case depth was determined to be the point at which the hardness stopped decreasing.

![Image](image1.png)

*Figure 10: Microhardness indentations through the case of a sample (2A).*

To analyze the data plots of the hardness indentations were generated in Excel so the trend of hardness values could be observed (Figure 12). For the sample used in Figure 12 the hardness became consistent at 450 microns deep in the sample, so the case depth was determined to be 450 microns.

![Image](image2.png)

*Figure 11: Measured hardness values vs depth in sample (2A).*
3. Results

There were three primary findings in this project. Understanding the kinetics of alpha case formation for samples undergoing repeat cycles at 1750°F one, two, three, or four times was the first goal of this project. A comparison of four, four hour cycles to a sixteen hour hold for any difference in case depth was also achieved. Finally, the effect of heating above the beta transus temperature and a 1350°F heating on alpha case depth was observed. These results are presented below.

3.1 Cycles 1-4

Alpha case depth measurement using each of the three measurement methods for the first four cycles and the sixteen hour hold are summarized in Table II. All measurement methods indicated that alpha case grows most rapidly during the first thermal cycle and the growth rate decreased with each further thermal cycle. Additionally, the microhardness data corroborates the visual estimation values. Visual analysis is both an accurate method of measuring alpha case and a fair representation of how alpha case affects the properties of the sample. Visual analysis is used to express the remainder of our results.

Table II: Summary of Alpha Case Depth for Cycles 1-4 and H Using All Analysis Methods

<table>
<thead>
<tr>
<th></th>
<th>Area Fraction</th>
<th>Visual Estimation</th>
<th>Microhardness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average (µm)</td>
<td>STDEV</td>
<td>Average (µm)</td>
</tr>
<tr>
<td>1</td>
<td>549</td>
<td>53</td>
<td>250</td>
</tr>
<tr>
<td>2</td>
<td>576</td>
<td>29</td>
<td>366</td>
</tr>
<tr>
<td>3</td>
<td>776</td>
<td>88</td>
<td>388</td>
</tr>
<tr>
<td>4</td>
<td>795</td>
<td>233</td>
<td>392</td>
</tr>
<tr>
<td>H</td>
<td>923</td>
<td>101</td>
<td>537</td>
</tr>
</tbody>
</table>

3.2 Four Cycles and Hold

The four cycle and hold samples measured using visual estimation had case depths of 392 and 537 microns respectively (Figure 12). For these samples using area fraction analysis a case depth of 795 and 923 microns was recorded. Four cycle and hold samples were compared using ANOVA with Tukey and Fisher pairwise comparisons. Both the visual estimations and area fraction methods showed no statistical difference in the four cycle and hold samples. The ANOVA meets all assumptions of normality and variances.
3.3 Beta Transus and 1350 °F

Table III and Figure 13 demonstrate the case depth values for the samples that experienced the beta transus temperature and the 1350°F heat treatment. This data indicated that the alpha case depth decreased in the four cycle and hold after the beta transus thermal cycle and again after the 1350°F thermal cycle. Using visual estimation, the alpha case depth on H and 4 samples were measured at 392 and 537 microns respectively. The 4β and Hβ decreased to 292 and 318 microns respectively and then to 263 and 281 after the 1350°F heating. The decrease in case depth after the beta transus and the 1350°F heating is attributed to material loss caused by excess scale flaking off. The glass coating normally used by Weber Metals forms a cohesive layer around the surface of the part. If used, this would have prevented the material loss seen after the beta transus and 1350°F thermal cycles.

Table III: Alpha Case Depth for 4, 4β, 4βT, H, Hβ, and HβT Using Visual Estimation

<table>
<thead>
<tr>
<th></th>
<th>Visual Estimation</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average</td>
<td>STDEV</td>
</tr>
<tr>
<td>4</td>
<td>392</td>
<td>112</td>
</tr>
<tr>
<td>H</td>
<td>537</td>
<td>139</td>
</tr>
<tr>
<td>4β</td>
<td>292</td>
<td>68</td>
</tr>
<tr>
<td>Hβ</td>
<td>318</td>
<td>37</td>
</tr>
<tr>
<td>4βT</td>
<td>263</td>
<td>19</td>
</tr>
<tr>
<td>HβT</td>
<td>281</td>
<td>48</td>
</tr>
</tbody>
</table>
4. Discussion

All three methods show the largest case growth during the first cycle. Subsequent cycles have a decreasing growth rate. This is expected due to the difference of oxygens diffusivity between the alpha and beta phases. Oxygen diffuses slower into alpha due to the differences in interstitial sites between the two phases. As more of the titanium is stabilized into the alpha phase oxygen is less able to diffuse towards the core, leading to the decreasing growth rates that we see in Figure 14.

![Graph of Alpha Case Depth for all Measurement Methods.](image)
Comparing the three methods of alpha case depth reveals that area fraction is overly sensitive with even a small increase in the percent area of the alpha phase is considered alpha case. This small increase, however, does not affect the properties. Alpha case should only be removed when it affects the properties of forged components. Microhardness traversal is the best method for quantitatively measuring where properties change and where the effective alpha case ends. The downside of this method is the time commitment to take numerous data points. Visual estimation values agreed with microhardness traversal values using a fraction of the time. After measuring the alpha case thickness by area fraction analysis, visual estimation, and microhardness traversal it was determined that visual estimation was the most efficient method of case thickness measurement. It was more accurate than the area fraction analysis, equal to the microhardness, but faster than the manual microhardness traversal method. Alpha case growth per cycle is depicted graphically in Figure 15. The amount of alpha that grew drastically decreases with each cycle. Using the visual estimate, an average case growth of 250 microns occurred during the first cycle, this decreased to 116 for the second cycle, 22 for the third, and 4 for the fourth. The standard deviation increased as the samples were subjected to more cycles. This is likely due to slight variances in temperature in the furnace. As more cycles are completed there is more opportunity for these variances to add together, leading to the larger error bars seen in Figure 15.

![Figure 15: The amount of alpha that grew during each of the first four cycles measured using the visual estimate. Note the decrease in the amount of new alpha case after subsequent cycles.](image.png)
5. Conclusions

1. Alpha case depth during the first cycle is 250 microns using visual estimation, and continues to 336 microns during the second cycle, 388 for the third, and 392 for the fourth with the growth rate leveling out.
2. Four cycles of four hours and one sixteen hour cycle are statistically similar per a Tukey and Fisher pairwise comparison test.

6. Recommendations

1. Remove alpha case once forging is complete rather than between forging steps to reduce cumulative material removal.
2. If available, use automatic microhardness to characterize depth, if not use visual estimations. Visual estimations closely match the change in properties associated with alpha case depth as measured by microhardness traversal.
References


