OPTIMIZING STRENGTH AND FRACTURE TOUGHNESS OF A CAST TITANIUM ALLOY THROUGH HEAT TREATMENT AND MICROSTRUCTURE CONTROL

A Thesis in
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by
Amy C. Robinson

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The thesis of Amy C. Robinson was reviewed and approved* by the following:

Donald Koss  
Professor Emeritus of Materials Science and Engineering  
Thesis Co-Advisor  
Co-Chair of Committee

Christopher Muhlstein  
Assistant Professor of Materials Science and Engineering  
Thesis Co-Advisor  
Co-Chair of Committee

Paul Howell  
Professor of Metallurgy

Clifford Lissenden  
Associate Professor of Engineering Science and Mechanics

Ernest Czyryca  
Senior Engineer, Carderock Division, Naval Surface Warfare Center  
Special Member

Gary Messing  
Professor of Materials Science and Engineering  
Head of the Department of Materials Science and Engineering

*Signatures are on file in the Graduate School
Abstract

The relationship between the microstructure and tensile ductility and fracture toughness for cast Ti-5111 was determined and compared to that of hot-rolled and annealed Ti-5111. Graphite mold cast Ti-5111 plate material was examined in the as-received condition and after six different heat treatments involving elevated temperature anneals followed by an air or furnace cool. Three investment cast Ti-5111 plates were also examined after annealing followed by either a fan cool, air cool, or furnace cool. All castings developed a lamellar colony microstructure consisting of aligned lamellae of alpha and beta phases. Altering the cooling rate from the annealing temperature had the most influence on the microstructure such that plates with a slower cooling rate typically developed coarser grain boundary alpha, larger alpha colonies, thicker alpha laths, and greater volume fractions of alpha phase. The average prior beta grain size for the graphite mold cast specimens ranged from 920 µm to 1360 µm, while that for the investment cast specimens was approximately 1750 µm.

The tensile behavior of the castings was characterized by a crack initiation and propagation process where the ductility was often limited by the strain required to initiate a large crack. The cracks formed along planar slip bands that crossed alpha colonies or in some cases, entire prior beta grains. Thus, reducing the alpha colony size and prior beta grain size should improve the casting ductility by limiting the length of slip-induced cracks. Due to the large grain and colony sizes present in the castings, the strength and ductility was observed to be sensitive to specimen size such that a smaller tensile diameter (i.e. 3.2 mm as compared to 12.5 mm)
decreased the tensile and yield strengths due to the high fraction of large grains located on the specimen surface that can yield by predominantly single slip. The scatter in ductility values in the smaller specimens was significantly greater as a result of fracture controlled by a crack initiation and propagation process within a single grain that comprises a large fraction of the specimen cross-section. Thus, once a large crack initiated, minimal additional strain was required to propagate the crack.

Both intrinsic and extrinsic toughening mechanisms were apparent in the fracture toughness study of the castings. The fracture initiation toughness was enhanced by secondary cracking and significant blunting at the crack tip as evidenced by the presence of strain-induced void formation within a large process zone. Large alpha colonies located at the transition between the fatigue pre-crack and tensile crack growth regions limited the fracture initiation toughness by promoting easy crack growth along a significant fraction of the crack front. Thus, limiting the alpha colony size should enhance the fracture initiation toughness. The best crack propagation resistance (tearing modulus) was observed from specimens with large alpha colonies and large prior beta grains. Enhancing the size of these features increased the surface roughness, and consequently the tearing modulus, due to greater crack deflection, crack bifurcation, and shear ligament toughening from the larger alpha colonies and prior beta grains. Crack bridging by the ductile beta phase was also observed and should enhance the tearing modulus. When compared to the hot-rolled and annealed plate, the graphite mold castings exhibited better fracture initiation toughness and crack propagation resistance.
However, the wrought plate maintained relatively good fracture initiation toughness and crack propagation resistance as a result of the continuous ductile beta matrix present in the microstructure.
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Chapter 1: Introduction

Titanium and titanium alloys are used in a variety of structural applications where weight savings and/or corrosion resistance are of particular interest. The U.S. Navy uses titanium structures and components in numerous ship and submarine applications due to their excellent combination of mechanical and physical properties. Titanium’s specific strength, high corrosion fatigue strength, good fracture toughness, excellent marine corrosion and erosion resistance, and weldability make it a desirable candidate for weight critical structural and machinery applications in shipbuilding. While the Ti-6Al-4V extra-low-interstitial alloy (ELI) is usually selected as the high-strength option for marine applications, there are concerns regarding its stress corrosion cracking resistance and fracture toughness, particularly under shock conditions.

The Ti-100 titanium alloy (Ti-6Al-2Cb-1Ta-0.8Mo) was developed and used by the U.S. Navy as an alternative for Ti-6Al-4V ELI. The Ti-100 alloy combined high strength (yield strength ≥ 690 MPa) and high toughness with good weldability making it suitable for hull material. However, the high cost of alloying elements, poor product yield, and negligible commercial market limited the use of this alloy.

The titanium alloy Ti-5111 (Ti-5Al-1Sn-1V-1Zr-0.8Mo) was developed as a lower cost, more producible alloy that is weldable and has strength/corrosion properties at least equivalent to Ti-100 [1]. The Ti-5111 alloy, which is a near-alpha alloy, was initially evaluated on the basis of production ingots that were melted, forged, and processed to plate, bar, forgings, and welding wire. This alloy, however, had never been evaluated in the cast form. By casting the Ti-5111 alloy into near
net shape components, numerous advantages are achieved, particularly significant schedule and cost savings. Today’s casting processes allow for the fabrication of near-net shape components even with complex geometries. By casting near-net shape components, secondary processing, such as machining and welding, is minimized. Less machining not only reduces time, but also material waste. Reducing welding procedures also dramatically reduces both time and cost of product fabrication. For example, the U.S. Army saved approximately 6000 hours of welding and 4000 pounds of titanium by casting the titanium components for the Howitzer [2].

Although Ti-5111 castings have the same chemistry as wrought products, their microstructures, and thus mechanical properties, differ due to the lack of mechanical deformation in the castings. Ti-5111 plate is usually hot-rolled in the beta phase field to produce a transformed beta structure. This microstructure consists of thick alpha plates surrounded by a transformed beta matrix. In turn, cast Ti-5111 consists of large prior beta grains outlined by grain boundary alpha, and an alpha colony structure consisting of thin, parallel alpha laths surrounded by thin layers of retained beta. These differences, discussed in more detail in Chapter 2, have significant effects on the fracture behavior, specifically on the location of void nucleation and growth, slip length, and crack propagation.

Prior to this study, no previous research or development had been conducted on cast Ti-5111. Based on experience with cast Ti-6-4, Ti-5111 was initially investment cast for Unmanned Underwater Vehicle (UUV) hulls. The heat treatments used for cast Ti-5111 were based on conditions developed solely for Ti-
6-4 castings, which include a final $\alpha/\beta$ anneal at 843 °C after casting and hot isostatically pressing (HIPing). Thus, cast Ti-5111 components were heat treated under the same conditions as cast Ti-6-4 due to foundry reluctance to risk heat treatments not within their standard practice. Preliminary testing on this casting revealed fracture toughness properties significantly below that of wrought Ti-5111 [3]. It stands to reason that because the alloys are chemically and structurally different, the optimum heat treatment for Ti-6-4 may not be applicable to Ti-5111, particularly when optimum strength and fracture toughness are desired. Results from studies on the heat treatment processes for Ti-5111 wrought products show optimum mechanical properties, particularly toughness, are achieved when the alloy is processed above the $\beta$-transus temperature of 980 °C and then subsequently annealed in the $\alpha/\beta$ field at 954 °C to produce a Widmanstatten alpha microstructure with retained beta phase [4,5]. The absence of mechanical deformation and differences in the heat treatment temperature and cooling rate for the cast product versus the wrought product will affect both the microstructure and the mechanical properties of the cast Ti-5111 alloy. Because the Ti-5111 alloy was developed by the Navy specifically to have high strength and fracture toughness, it is essential to understand the microstructure development and fracture processes in the cast product to ensure these mechanical properties are met in this lower cost form.

A fundamental knowledge of the microstructure development in cast near alpha titanium alloys, and specifically Ti-5111, is currently limited. The literature describes only a few studies that examine the influence of microstructure on the mechanical properties of cast near-alpha or alpha-beta titanium alloys, and of those,
the majority focus on Ti-6-4 and similar alpha-beta alloys [6,7,8,9]. Additionally, much of the focus on cast Ti-6-4 is for dental and medical devices and various aerospace applications. Therefore, the mechanical properties of interest are predominantly strength, ductility, and fatigue; not fracture toughness [10,11,12]. The lack of knowledge on microstructure development and fracture behavior of cast near-alpha titanium alloys limits their use in structural applications where strength and fracture toughness are critical properties.

This study aims to develop a scientific foundation correlating the effect of heat treatment conditions on the microstructure development and resulting tensile and fracture toughness properties of cast Ti-5111. A primary goal of this research is to understand the fundamental mechanisms of fracture in Ti-5111 castings in terms of the controlling features of the microstructure, specifically the alpha and beta phases and their morphology. Attention is given to the role of the effective slip length (directly related to the prior beta grain size, alpha colony size, alpha lath thickness, and grain boundary alpha thickness) and its ability to initiate and propagate cracks. By understanding the basic mechanisms of fracture in cast Ti-5111, engineers can optimize strength and fracture toughness properties for use in military and commercial applications, including unmanned underwater vehicle hulls, piping, valves, pumps, and other seawater applications. In order to understand better the role of microstructure in controlling the properties of Ti-5111, the wrought product form of Ti-5111, specifically in the hot-rolled and annealed condition, will also be examined to identify the causes for differences in their tensile and toughness properties.
Chapter 2: Background & Literature Review

In order to understand the fundamental fracture mechanisms in cast Ti-5111 and to optimize its strength and fracture toughness, it is first necessary to understand the phase transformations that can occur in this alloy and the microstructural features that influence the material’s strength and fracture toughness.

2.1 Phase Transformations

Titanium alloys can be classified into four categories: alpha (α), near-alpha, alpha-beta (α/β), and beta (β) [13,14]. An alloy is classified in a specific category based on its composition and typical phases present. The Ti-5111 alloy is considered a near-alpha alloy due to its high concentration of alpha stabilizers (5 wt% aluminum), which cause it to typically develop a Widmanstatten alpha structure. The alpha phase exhibits a hexagonal close packed (hcp) crystal structure at room temperature, while the beta phase exhibits a body centered cubic (bcc) structure. A Ti-Al binary phase diagram is shown in Figure 1a while a magnified region of the α/β region from a Ti-beta stabilizer phase diagram is illustrated in Figure 1b [13].

The Ti-5111 alloy composition falls between the more common Ti-8Al-1Mo-1V and Ti-6Al-2Sn-4Zr-2Mo alloys, and its β-transus is 980 °C [4,15]. The ASTM chemistry for Ti-5111 is given in Table 1. As described elsewhere [4,16], the alpha soluble elements include aluminum (Al), oxygen (O), nitrogen (N), and carbon (C), which were added to stabilize the alpha phase and increase strength. Tin (Sn) and zirconium (Zr) were added for increased strength and are considered neutral.
elements because they are soluble in both the alpha and beta phases. Silicon (Si) was added to enhance creep resistance, strength, and fracture toughness. Zirconium not only increases strength, but also helps promote a uniform silicide distribution in the matrix material. Finally, the β-stabilizers, molybdenum (Mo) and vanadium (V), produce a small increase in strength while Mo significantly improves toughness. Oxygen (O) is limited to 0.11 weight percent maximum due to its deleterious effects on fracture toughness at higher levels.
Figure 1: Binary titanium phase diagrams for (a) titanium – aluminum and (b) titanium – beta stabilizer
Table 1: Chemical Composition of Ti-5111 (weight percent)

<table>
<thead>
<tr>
<th>Element</th>
<th>ASTM B 265 Grade 32</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>4.5-5.5</td>
</tr>
<tr>
<td>Tin</td>
<td>0.6-1.4</td>
</tr>
<tr>
<td>Vanadium</td>
<td>0.6-1.4</td>
</tr>
<tr>
<td>Zirconium</td>
<td>0.6-1.4</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>0.6-1.2</td>
</tr>
<tr>
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<td>0.25 max</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.06-0.14</td>
</tr>
<tr>
<td>Oxygen</td>
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</tr>
<tr>
<td>Carbon</td>
<td>0.08 max</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>0.03 max</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>0.015 max</td>
</tr>
</tbody>
</table>

During cooling of the Ti-5111 from the beta phase region, the metastable beta transforms to alpha through a nucleation and growth process. Alpha-phase precipitates nucleate along the beta-phase grain boundaries and grow in such a manner that their basal plane is parallel to the (110) plane of the beta grain [14,17]. As the alpha precipitates continue to grow, their broad side grows slowly due to their coherency with the matrix, while the edge sides (incoherent with respect to the matrix) grow more quickly forming elongated plates [17,18].
While all cast Ti-5111 products have a transformed beta structure, there can be important differences depending on the temperature of solution treating and the subsequent cooling rate. The alpha plates can develop in well-defined colonies nucleating from the grain boundaries, or they can develop as individual laths (basketweave $\alpha$) independent of the grain boundaries. The lath scale can range from fine to coarse, and they may be continuous or discontinuous. The alpha laths are surrounded by thin (~<0.1$\mu$m) layers of retained beta, which is enriched in $\beta$-stabilizing elements \cite{18}. The layers of retained beta phase form as a result of diffusion of the $\beta$-stabilizing elements ahead of the migrating alpha interface. Additional microstructure features affected by the solution heat-treat temperature and cooling rate include: prior beta grain size, alpha colony size, grain boundary alpha thickness, and the volume fraction of the alpha and beta phases. Each of these microstructure features are controlled by the time and temperature of the heat treatment, as well as the cooling rate. In order to optimize both strength and fracture toughness, the effect from each of these features must be well understood and controlled.

2.2 Microstructure Development in Wrought Hot-Rolled Ti-5111

The wrought Ti-5111 alloy is different from other near-alpha titanium alloys due to its combination of strength, high fracture toughness, and stress corrosion cracking resistance, which are critical properties for Navy structural materials. The Ti-6Al-4V ELI (extra low interstitial) alloy is a typical alloy considered for high-strength marine applications. However, its fracture toughness and stress corrosion
cracking resistance are insufficient for critical applications, particularly under shock conditions [1]. Similarly, the majority of near-alpha titanium alloys do not have the combination of high strength, fracture toughness, and stress corrosion cracking resistance as the Ti-5111 alloy does.

An important difference between the wrought Ti-5111 plate examined in this study and similar near-alpha titanium alloys developed for high fracture toughness is the processing steps and subsequent microstructure developed. Typically, commercial α+β titanium alloys requiring good fracture toughness have "β-annealed" structures. These structures are developed by homogenizing in the β phase field, deforming in the α/β region, recrystallizing above the β transus, and cooling at a slow to moderate rate, followed by stress-relieving in the α/β region [19]. The resulting microstructure is fully lamellar, similar to that shown in Figure 2. Lamellar microstructures are characterized by the parallel alpha laths (light regions) developed in well-defined colonies with grain boundary alpha phase decorating the prior beta grain boundaries. The alpha laths are surrounded by a thin (sub micron) layer of retained beta phase (dark regions). The lamellar microstructure shown in Figure 2 is similar to the starting ingot cast structure present prior to deformation.

The wrought Ti-5111 plate examined in this study was homogenized in the β-phase field with subsequent rolling beginning in the beta phase region and concluding in the α/β phase region. The material was then given an α/β anneal at 954 °C followed by an air-cool. This type of processing produces a "β-processed" condition not extensively used for α+β alloys in commercial applications [14]. Beta-processed alloys do not undergo a recrystallization anneal after deformation.
Instead, they are only annealed below the beta transus [14,20,21,22]. This β-processing produces a deformed Widmanstatten alpha microstructure, as shown in Figure 3 and Figure 4. The micrograph in Figure 3, taken parallel to the rolling direction of the plate, shows some globular alpha phase in a matrix of fine acicular alpha and retained beta. In Figure 4 (transverse direction), the alpha appears as thick, elongated plates with some parallel alignment between several groups of plates. A minimal amount of broken-up retained grain boundary alpha phase is also evident in the micrograph.

Commonly, near-alpha and α/β alloys requiring high strength are rolled in the α/β region followed by an α/β anneal to produce a bi-modal microstructure of equiaxed alpha and transformed beta. The microstructure tends to produce the best tensile strength and ductility [9,19,20], but its fracture toughness is inferior to that of a beta annealed microstructure. As a result, for superior fracture toughness a Widmanstatten α + β structure is desirable. This microstructure has been shown to increase fracture toughness by causing more crack deflection [23,24,25,26], a phenomenon further investigated in the following section.
Figure 2: Typical lamellar microstructure developed from hot-rolled plate after a recrystallization anneal in the β phase region (Kroll’s etchant)

Figure 3: Widmanstatten microstructure of hot-rolled Ti-5111 (parallel to rolling direction, Kroll’s etchant)
The majority of the strength in Ti-5111 is obtained by solid solution strengthening, as is typical for near alpha titanium alloys [13,14,17]. This type of strengthening can be accomplished by the addition of either substitutional elements or interstitial elements. The primary strengthener in Ti-5111 is aluminum. Aluminum is a substitutional element that replaces the titanium atoms while maintaining the hcp crystal structure. Aluminum atoms are slightly smaller in size than the titanium atoms; therefore, a contraction of the lattice occurs, strengthening the material.
The second type of solid solution strengthening in Ti-5111 is from interstitial atoms such as oxygen, nitrogen, and carbon. These interstitial elements strain the hcp lattice through expansion because they sit between titanium lattice sites. In small concentrations these elements are excellent strengtheners; however, at high levels they become deleterious to the ductility and fracture toughness. Given all titanium strengthening elements, nitrogen is the most potent strengthener. However, oxygen additions are most often made because they are simpler and oxygen is still an effective strengthener. The oxygen content is kept to a maximum of 0.11 weight percent to retain high fracture toughness.

Although solid solution strengthening is the primary method for strengthening near-alpha alloys, it is possible to make minor strength variations by altering the microstructure. For the hot-rolled plate examined in this study, the critical microstructure features include the alpha platelet width, alignment of the alpha plates (parallel or acicular), volume fraction of retained beta, and the structure within the transformed beta matrix. In general, finer alpha laths increase strength by decreasing the material slip length [21,22]. The decrease in slip length leads to larger dislocation pile-ups at the alpha lath / retained beta interfaces; thereby increasing strength [27].

Studies of the influence of microstructure on the strength and fracture toughness of cast near alpha titanium alloys are rather limited, and of these, only a few alloys have been examined, such as the α/β alloys Ti-6-4 and IMI 550 (Ti-4Al-4Mo-2Sn-0.5Si) [6,7,8]. These studies report the alloys in the cast form have fracture toughness properties superior to their wrought product form due to the
lamellar microstructure developed. As Kearns et al. report [9], castings with a coarse lamellar structure provide the best fracture toughness.

Because the literature is limited on its discussion of the influence of microstructure on the strength and fracture toughness of cast near alpha titanium alloys, studies regarding microstructure influence on these properties for wrought titanium alloys are reviewed below. As discussed previously, wrought near-alpha and alpha + beta titanium alloys can be “β-annealed” to develop lamellar microstructures, which have been shown to promote good fracture toughness properties due to crack deflection from the alpha laths [28,29,30,31,32,33]. Nevertheless, the particular microstructure feature controlling fracture toughness differs among authors. Critical features include (1) prior beta grain size, (2) alpha colony size, (3) alpha lath thickness, (4) grain boundary alpha thickness, or (5) a combination of any or all of these features.

Studies conducted by Greenfield and Margolin [34,35] and Padkin et al [36] on Ti-5.25Al-5.5V-0.9Fe-0.5Cu showed fracture toughness increased as the volume fraction and thickness of the grain boundary alpha phase increased. The fracture toughness increased linearly by 27 MPa-m^1/2 as the grain boundary alpha thickness increased from 2.5 μm to 5.5 μm. Thicker grain boundary alpha had no further influence on the fracture toughness. The authors theorized that the increase in fracture toughness was achieved by the ability of the softer grain boundary alpha to deform plastically and absorb more energy than the harder surrounding transformed beta matrix. However, Chesnutt et al. [37] showed the presence of grain boundary alpha decreased fracture toughness due to unstable fracture occurring along this
softer phase. The increase in grain boundary alpha phase led to intergranular fracture along the grain boundaries. In accordance with this observation, they suggest a smaller prior beta grain size would improve fracture toughness because larger prior beta grains tended to have more continuous alpha phase situated on the grain boundaries. A similar study by Rogers [28] was conducted on the near-alpha alloy Ti-6Al-5Zr-0.5Mo-0.25Si and the alpha+beta alloy Ti-6Al-4V; however, it showed no significant relationship between fracture toughness, grain boundary alpha thickness, and prior beta grain size.

The thickness of the alpha laths and the size of the alpha colonies may also play an important role in controlling fracture toughness. Studies by Boyer et al. [38] on Ti-6-4 and Hall et al. [39] have shown that thicker alpha laths lead to numerous changes in crack propagation direction, which results in an increased crack path length and correspondingly greater energy absorption. An increase of 13 MPa-m$^{1/2}$ in fracture toughness was observed in Ti-6-4 by increasing the alpha lath thickness from 1.5 µm to 4 µm [38]. Additionally, Hall and Hammond [30,39] suggest higher lath aspect ratios lead to more layers of retained beta that act as crack arrestors. The retained beta phase is believed to be more ductile than the alpha phase, and thus, it leads to an increase in fracture toughness. Finally, Chan et al. [40] suggest the fracture toughness of lamellar TiAl alloys increase with increasing colony size because of the tendency to form larger crack-wake ligaments in coarse-grained materials, such as castings. However, in the above mentioned study by Boyer et al. [38], no relationship was found between colony size and fracture toughness.
As these studies show, very little agreement exists among the authors as to what microstructure features control fracture toughness in near-alpha and alpha + beta titanium alloys. Although a lamellar microstructure has been proven to enhance fracture toughness, the controlling features of that structure are still unknown. By examining the influence of microstructure on the strength and fracture toughness of cast Ti-5111, this study will provide a better understanding of the controlling features of the lamellar microstructure on fracture toughness of this alloy in the cast condition.

The effect of microstructure features on the strength of near-alpha and alpha + beta titanium alloys is more straightforward. An inverse relationship between strength and fracture toughness exists in titanium alloys [38,41,42,43]. As noted previously, thicker alpha laths (developed by slower cooling rates) enhances fracture toughness, while fine alpha laths (developed by increasing cooling rates) increase strength [21,22,44,45]. The increase in strength from fine alpha laths is attributed to decreased slip length [21,22,45]. Thinner laths have a much smaller slip length and may increase the material's strength.

Lütjering [22] reported alpha colony size plays the most significant role in controlling tensile strength and ductility because it determines the effective slip length in an alloy. As the cooling rate increases, the colony size and slip length decreases, and thus, the yield strength increases. Gerberich and Baker [45] also agree that decreasing colony size leads to increased strength [48]. Williams et. al [42] showed that voids tend to nucleate at alpha plate / beta interface boundaries. Therefore, long alpha plates (large colonies) were believed to provide a greater area
for void nucleation and growth, thereby limiting the strength and ductility in lamellar titanium alloys.

The presence of grain boundary alpha may reduce both strength and ductility. When a continuous layer of grain boundary alpha exists, Greenfield et al. [44] believe only one void needs to nucleate along the grain boundary to cause complete failure to occur. Additionally, Sauer and Lütjering [46] and Williams et al. [42] found that a thick and continuous grain boundary alpha layer can reduce ductility in titanium alloys due to crack propagation along the soft grain boundary alpha phase. They have shown that in Ti-6Al-2Sn-4Zr-6Mo the strength difference between the weaker grain boundary alpha layers and the higher strength beta matrix cause strain concentrations and void nucleation along the alpha phase.

Although the effect of microstructure on the strength of near-α and α+β titanium alloys is better understood than that of fracture toughness, the above discussion illustrates that the controlling feature is still controversial, especially under conditions where the fracture path is not along a prior beta grain. In addition to determining the role of microstructure on the fracture toughness of cast Ti-5111, this study will also determine the controlling microstructure feature on strength of cast Ti-5111.

2.4 Microstructure Differences between Wrought and Cast Ti-5111

In the as-cast condition, the Ti-5111 alloy is effectively cooled from the β phase field down through the α/β phase field to produce a transformed beta microstructure. The casting is then typically hot isostatically pressed at 900 °C to
close residual porosity, and subsequently cooled at a rate equivalent to an industry air-cool to produce a lamellar microstructure as that shown in Figure 5. The alpha laths develop in well-defined colonies with continuous grain boundary alpha outlining the prior beta grains.

The microstructure development in hot rolled and annealed Ti-5111 plate is significantly different from that of cast Ti-5111 due to the deformation process and subsequent $\alpha/\beta$ anneal. As discussed in Section 2.2, hot-rolling of Ti-5111 plate begins in the beta phase region and finishes in the alpha + beta region. The starting microstructure of the material to be hot rolled looks identical to the original cast structure. However, during rolling in the beta phase field, the large prior beta grains deform significantly and often break down. Subsequently, the plate is annealed in the $\alpha/\beta$ field and air-cooled to produce a transformed beta structure of coarse Widmanstatten alpha plates surrounded by a beta matrix, as shown in Figure 6. The beta matrix consists of very fine (submicron) alpha laths in a basketweave orientation surrounded by retained beta.

Due to the significant difference in microstructure between the cast and wrought Ti-5111 alloy, the strength and fracture toughness will be different. With respect to the microstructure features that control the strength and fracture toughness of hot rolled and annealed Ti-5111 plate, the alpha plate size is the dominating feature. As with the alpha lath thickness in castings, the alpha plate thickness in the wrought material controls the slip length. The alpha plates in the hot-rolled plate are much thicker and shorter than the laths developed in the
castings; therefore, the slip length through the width of the alpha plate is larger in the hot-rolled plate.

Another significant difference in the microstructures, and thus resulting properties, between the cast and hot rolled Ti-5111 product is the presence of grain boundary alpha phase in the cast product. As the casting cools below the beta transus, alpha phase nucleates along the prior beta grain boundaries. As discussed earlier, the presence of the “weaker” grain boundary alpha phase may cause voids to nucleate and grow more easily than in the wrought material that lacks the grain boundary alpha. Thus, theoretically, the cast structure may have lower tensile strength and ductility than the wrought plate [9,42,46].

Another reason for expected lower strengths in cast Ti-5111 as opposed to the wrought plate is the presence of well-defined alpha colonies and large prior beta grains in the cast plates. These colonies and large grains greatly increase the slip length in the material. As Lutjering et al [14,22] have shown, a larger slip length decreases material strength as well as ductility.

In summary, the cast plate develops a lamellar microstructure consisting of large prior beta grains outlined with grain boundary alpha. Inside the prior beta grains are alpha colonies comprised of aligned alpha laths. In contrast, the hot-rolled Ti-5111 plate consists of thick Widmanstatten alpha plates within a transformed beta matrix, which consists of fine acicular alpha and retained beta. The wrought microstructure has no remaining evidence of the prior beta grains or grain boundary alpha. These significant differences in microstructure will lead to a difference in the strength, ductility, and fracture toughness of cast Ti-5111.
Figure 5: Typical as-cast and HIPed microstructure of Ti-5111 (Kroll's etchant)

Figure 6: Microstructure of hot-rolled and annealed Ti-5111 plate (Kroll's etchant)
2.5 Investment Casting vs. Graphite Mold Casting

Casting techniques have developed and improved greatly over the years. Although traditional casting methods such as graphite mold casting still exist and are very useful for certain applications, a casting technique called “investment casting” has enabled the production of near net-shape titanium alloy components (i.e. Ti-6Al-4V and Ti-4Al-4Mo-2Sn-0.5Si) with rather complex geometries [7,9]. The Navy intends to use the cast Ti-5111 alloy for applications that require both graphite mold castings and investment castings. Due to the differences between these casting techniques, such as mold material, mold thickness, and mold pre-heats, microstructure differences can occur. This section describes each of these casting techniques and their advantages and disadvantages.

As the name implies, graphite mold casting uses a graphite mold to make the titanium casting. In this process, an initial pattern mold made of wood or fiberglass is developed. Next, graphite powder is mixed with an epoxy, poured into the wood or fiberglass pattern, and mechanically rammed against the pattern to form a mold. The graphite mold is then removed from the outer pattern and dried to ensure it has the strength necessary to hold the molten titanium metal. After the metal is poured and cooled, the mold is removed by breaking it mechanically. The graphite mold needs to be a minimum of 5 mm thick and is in the form of a rather simple geometry [47]. This type of casting mold is best suited for large, thick castings that do not require tight dimensional tolerances to be maintained.

The investment casting process is similar to graphite mold casting with a few key differences. First, an inverse pattern of the final part is constructed. Next, wax
is poured into this inverse pattern taking the shape of the final component. The wax pattern is dipped into a ceramic slurry consisting of sand, zirconia, yittria, and other binding agents. The pattern is dipped and dried repeatedly until a ceramic shell at least 1 mm thick is formed. The shell is then fired to melt the wax and dry the ceramic, giving it the strength necessary to hold the molten titanium. Finally, the shell is placed in a vacuum, preheated, and titanium is poured into it. For each casting method, once the casting cools to below ~538 °C, the casting is removed from the vacuum furnace and the mold is mechanically removed. The casting is then HIPed to eliminate porosity and given an acid bath to remove any oxidation on the surface of the casting. The advantages of investment casting include the ability to fabricate complex geometries and produce castings of tight dimensional tolerances. This technique can only be used for thin castings less than 7.5 cm thick, and it is more expensive than the graphite mold method.

Typically, investment cast molds must be preheated to higher temperatures than graphite mold castings to increase molten metal flow [47]. The result of the higher pre-heat temperature will cause the casting to remain above the beta transus temperature for a longer period of time, leading to larger prior beta grain sizes. Additionally, the cooling rate between molds will differ due to the thermal conductivity difference of the graphite mold wall and the ceramic shell. The difference in wall thickness between each mold is also significantly different and will affect the cooling rate of the casting. Although an in-depth scientific investigation on the effects of graphite mold casting versus investment casting is outside the scope of this study, a brief discussion on the role of cooling rate between the castings will
be presented when discussing the prior beta grain size developed from each casting method.
Chapter 3: Experimental Procedures

3.1 Materials

3.1.1 Casting

For this study, the titanium alloy Ti-5111 was examined in two different cast forms: investment cast (IC) and graphite mold cast (GMC). The chemistry of each casting, along with the ASTM Standard B265 Grade 32 for Ti-5111 is shown in Table 2. PCC-Structurals, Inc. investment cast two plates 25 mm-thick (Heat # H4654) and subsequently hot isostatically pressed (HIPed) them at 900 °C and 103 MPa for two hours. The precise cooling rate of the casting from the pour is unknown; however, it is estimated to be around 25 °C/min, and the cooling rate after HIPing was approximately 2 °C/min. The plates were cut into three separate pieces (labeled IC 1, IC 2, and IC 3) and heat-treated according to Table 3. Additionally, Wah Chang cast two 32 mm-thick plates using a graphite mold (Heat # R286) with a cooling rate of approximately 45 °C/min. The plates were subsequently HIPed by PCC Structural, Inc. under the same conditions as the investment cast plates, cut into several smaller plates (GMC 1, GMC 2, GMC 3, GMC 4, GMC 5, GMC 6, and GMC 7), and heat-treated according to Table 3.

At the onset of this study, castings IC 1 and IC 3 were examined and tested to determine general microstructure and selected mechanical properties of cast Ti-5111. After this initial study, the heat treatments for the graphite mold castings as well as IC 2 were then developed based upon the knowledge gained from IC 1 and
IC 3. As will be discussed later, each of these heat treatments was designed specifically to vary critical microstructure features that correlate to the material’s strength and toughness. The cast + HIP plate condition from the graphite mold casting is used as a benchmark for each of the other heat treatments. The remaining six heat treatments for the graphite mold casting vary the solution treatment temperature (phase region) and the cooling rates from each temperature. All plates were held at temperature for 1 hour before being cooled to room temperature. For the duplex anneals (both a beta and alpha+beta anneal), the plates were heated into the beta regime, held for 1 hour, cooled to room temperature, and then heated to the two-phase alpha+beta regime, held for 1 hour, and again cooled to room temperature. A cooling rate of 1 °C/min is roughly equivalent to an industry furnace cool, 14 °C/min is approximately equivalent to an air cool, and 55 °C/min corresponds to a fan cool.
Table 2: Chemical Composition of Ti-5111 in Weight %

<table>
<thead>
<tr>
<th>Element</th>
<th>ASTM B 265 Grade 32</th>
<th>Investment Casting</th>
<th>Graphite Mold Casting</th>
<th>Hot-rolled Plate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
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<td>5.01</td>
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<td>1.02</td>
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<td>Iron</td>
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Table 3: Thermal Processing Parameters for Ti-5111 Castings

<table>
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<tr>
<th>Plate ID</th>
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<th>Cooling Rate (°C/min)</th>
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<td>IC 1</td>
<td>investment</td>
<td>β</td>
<td>1010</td>
<td>55</td>
</tr>
<tr>
<td>IC 2</td>
<td>investment</td>
<td>β, α+β</td>
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<td>55, 14</td>
</tr>
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<td>IC 3</td>
<td>investment</td>
<td>α+β</td>
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<td>1</td>
</tr>
<tr>
<td>GMC 1</td>
<td>graphite mold</td>
<td>cast+HIP</td>
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<td>2</td>
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<tr>
<td>GMC 2</td>
<td>graphite mold</td>
<td>β</td>
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<td>14</td>
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<td>GMC 3</td>
<td>graphite mold</td>
<td>β</td>
<td>1010</td>
<td>1</td>
</tr>
<tr>
<td>GMC 4</td>
<td>graphite mold</td>
<td>β, α+β</td>
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<td>graphite mold</td>
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</tbody>
</table>

3.1.2 Hot-rolled Plate

Titanium Metals Corporation (TIMET) produced a 4500 kg ingot of Ti-5111 that was converted to a 25 mm thick plate by hot rolling above the β-transus temperature of 980 °C, air cooling to room temperature, and annealing in the α+β
phase field for one hour at 954 °C, followed by an air cool. The chemical composition of the hot rolled plate is given in Table 2. The focus of this thesis was to study the effect of heat treatment conditions on the structure and properties of cast Ti-5111 plate; therefore, only the industry standard practice heat treatment was considered for the hot-rolled (HR) plate. However, as a benchmark for behavior, the hot-rolled material properties will be compared to that of the IC and GMC properties.

3.2 Microstructure Characterization

3.2.1 Castings

Metallography was conducted on all casting conditions to characterize the microstructure developed based on casting method (investment vs. graphite mold) and subsequent heat treatments. For each casting, (a) prior beta grain size, (b) alpha colony size, (c) alpha lath thickness, (d) grain boundary alpha thickness, and (e) volume fraction of the alpha and beta phases was determined. Sample preparation included hot mounting, grinding, and polishing using 6 µm, 3 µm, and 1 µm diamond slurries. The samples were etched by swabbing them with Kroll’s etchant for approximately 10 seconds to reveal the alpha and beta phases. Kroll’s etchant is made of 1 mL HF, 2 mL HNO₃, 50 mL H₂O₂, and 20 mL of H₂O. Specimens were examined under white light as well as polarized light, which reveals each individual colony as a different color based on its orientation.

Due to the complicated microstructure developed in titanium alloys with a transformed Widmanstatten structure, quantifying microstructure features can pose
a significant challenge. The following sections describe the techniques used to measure critical microstructure features in each of the castings. The methods used are based on ASTM standards, methods developed by one of the leading research groups in quantitative titanium microscopy [48,49], and variations of these methods as deemed appropriate for the current situation. Neither the ASTM standards nor techniques developed by Searles et al. [48,49] could be used alone due to limitations of their processes.

3.2.1.1 Prior Beta Grain Size

Due to the colony structure in this alloy, typical automated grain size measurement techniques could not be used to measure the prior beta grain size in these castings. Instead, the grain sizes were calculated manually using the Planimetric Procedure outlined in ASTM E 112 [50]. Using this procedure, a micrograph of known area is captured, and the grains within that microstructure are individually counted such that all full grains are counted as one and any grains intersecting the edge of the image are counted as one-half. The total number of grains is divided by the area measured. This value can then be converted into a grain size corresponding to the mean linear intercept using Table 1 of ASTM E 112. Because the prior grain size was on the scale of 1 mm, an area approximately 50 x 40 mm in size was measured for each casting condition.

3.2.1.2 Alpha Colony Size

The alpha colony size was measured for each casting condition using a variation of the linear intercept method described in ASTM E 112. In general, a set
of random lines was overlaid on a microstructure image, and wherever a line
intercepted a colony boundary it was segmented (erased) such that the remaining
line lengths were a measurement of the colony size. An illustration of the set of
random lines over an actual Ti-5111 casting micrograph is shown in Figure 7. The
intersection of each line with a colony boundary was marked manually, and Clemex
Vision© software was subsequently used to automatically calculate the remaining
line segments. This technique is the same as used by Searles and Tiley et al.
[48,49] for measuring alpha colony size of titanium alloy Ti-6Al-4V.

Although this method was determined by Searles and Tiley et al. [48,49] to be
the most accurate for measuring alpha colony size, there exists two notable issues
with this method. First, the choice of magnification to measure colony size is difficult
due to the large range in colony size. The colonies measured in these castings
range from only a few microns to over a millimeter in scale. This large range in size
makes it difficult to use a magnification that is high enough to allow one to
distinguish small colonies while being low enough to allow the measurement of
several whole, large colonies in one image. The number of measurements per
micrograph ranged from 20 – 75, depending on the colony sizes. For each casting,
200 – 300 measurements were taken to obtain an average colony size. A “colony”
was arbitrarily determined to have at least five parallel laths to be measured as a
separate colony.

Second, occasionally a large colony will be intersected by a much smaller
colony. Because we are measuring three-dimensional colonies using two-
dimensional images, it is impossible to know exactly how these colonies meet in
three dimensions. When a large colony is intersected by a smaller colony (Figure 8), the average colony size may be skewed smaller than reality because the large colony is now measured as two smaller colonies.

Figure 7: Random line pattern used to measure alpha colony size
3.2.1.3 Alpha Lath Thickness

Due to the different cooling rates employed after heat-treating, the castings develop alpha laths of varying thickness. To measure alpha lath thickness, Clemex Vision® software was utilized using a variation of the linear intercept method from ASTM E 112. The measurement process first involved capturing a micrograph image at a magnification between 500X and 4000X, depending on lath thickness. The majority of the specimens were imaged with the optical microscope. However, for specimens with a very fine lath thickness, such that a magnification greater than 1000X was necessary to delineate laths, images were captured using a scanning electron microscope and subsequently analyzed with the software. For both imaging procedures, once the image is captured, a series of concentric ovals are overlaid on...
the image, as shown in Figure 9. The image is then thresholded to a red and black color scheme (red = alpha, black = beta) for the computer to recognize the alpha lath boundaries. Similar to the measurement of the alpha colony size, wherever the ovals intersect a lath boundary, the line is “erased” and the remaining line segments are automatically measured, as shown in Figure 10.

For each specimen, 15 – 25 fields-of-view were measured such that a total of approximately 6000 measurements were made for each specimen. This large number of measurements was taken to ensure the most accurate average lath thickness was measured.

Quantitative stereology procedures were employed to measure the alpha lath thickness as accurately as possible. As described in the stereology book by Underwood [51], the distance between lamellae (or laths for titanium) can be represented as $\delta_t$ for true distance, $\delta_a$ for apparent distance, and $\delta_r$ for random distance, as illustrated in Figure 11. Because it is virtually impossible to measure $\delta_t$, the $\delta_r$ is typically measured and reported in the literature. To measure $\delta_r$, a set of lines are overlaid on a microstructure such that they intersect the laths at random orientations. The mean linear intercept is then calculated, and this value is equivalent to the mean random distance, $\bar{\delta_r}$.

The choice of location for each field-of-view on the sample was random unless: 1) the field included grain boundary alpha phase, or 2) alpha laths intersected the surface at a large oblique angle such that the laths were more parallel to the surface than perpendicular. An example of a region with alpha laths intersecting the surface at a large oblique angle is shown in Figure 12. These laths
are tilted such that a three-dimensional view of the beta phase can be seen due to etching. These regions were avoided for the alpha lath measurements.

Figure 9: Optical micrograph of alpha laths overlaid with concentric ovals

Figure 10: Remaining line segments used to measure the alpha lath thickness
Figure 11: Schematic illustrating the measurement of lamellae (lath) thickness in lamellar structures (adapted from Underwood [51])
3.2.1.4 Volume Fraction of Alpha and Beta Phases

Measurement of the volume fraction of alpha phase for each casting used optical microscopy and the Clemex Vision© software. Similar to the alpha lath measurements, a microstructure image was captured and thresholded such that the alpha phase was colored red and the beta phase was colored black. The software is then able to calculate the fraction of the image that is red, correlating to the amount of alpha phase in each casting. These images were captured at the lowest magnification possible (typically 500X) while still being able to delineate alpha laths. One hundred fields-of-view were measured over a 10 x 10 square grid pattern for each specimen. Both alpha colony regions and grain boundary alpha were included in these measurements.

Figure 12: Example micrograph showing alpha laths intersecting the surface at an oblique angle
3.2.1.5 Compositional Analysis of Plate and Alpha and Beta Phases

To ensure the castings were chemically homogeneous through the thickness of each casting, specimens from the investment and graphite mold castings were analyzed via electron probe X-ray microanalysis (EPMA). This technique provides elemental analysis by bombarding a specimen with electrons to produce characteristic X-rays. For both the investment casting and graphite mold casting, two heat treatment conditions (IC 1 and IC 2 and GMC 1 and GMC 2, respectively) were sectioned such that the through thickness composition could be determined. Due to specimen size requirements, two specimens for each plate had to be measured. To ensure no loss of data, the center of each casting was measured on both specimens “halves” leading to some overlap in data and measurements across approximately 35 mm for each casting. Samples were prepared by polishing with 0.3 μm colloidal silica solution. A Cameca SX50 instrument was used to obtain the EPMA data with measurements taken every 40 μm to obtain compositional averaging over both the alpha and beta phases. The elements detected included Al, V, Sn, Zr, Mo, Si, and Fe.

The compositions of the individual alpha and beta phases were also determined using EPMA. A spot size of 1 μm was used to measure each phase. The alpha phase was thick enough to obtain data solely from the alpha phase; however, for the beta measurements, some signal from the alpha phase was measured due to the fine scale of the beta phase. Wherever possible, optically observed regions of thicker beta phase were used to take the measurements.
3.2.2 Hot-rolled Plate

The critical microstructure features in the hot-rolled and annealed Ti-5111 plate include (a) alpha plate thickness and (b) volume fraction of the alpha and beta phases. The alpha plate thickness measurements were conducted in a similar manner as described in Section 3.2.1.3 Alpha Lath Thickness for castings using the line intercept method. The volume fraction of the alpha and beta phases was also measured in the same manner as for cast Ti-5111; see Section 3.2.1.4 for details.

3.3 Mechanical Testing

3.3.1 Castings

3.3.1.1 Hardness

Hardness measurements were taken for all ten casting conditions. Measurements were made using a Rockwell C hardness tester. A minimum of three measurements was taken on each sample, and the average of these values is reported.

3.3.1.2 Tensile

Three tensile specimens with a diameter of 6.4 mm and a gage length of 25.4 mm (Figure 13) were initially tested for each of the casting conditions in accordance with ASTM E 8 [52]. These tests were used to determine the yield strength, ultimate tensile strength, elongation, and reduction in area of the castings. The tests were conducted at a strain rate of $10^{-3} /s$ and at the ambient laboratory temperature (~ 20
A extensometer was used to measure strain during the test. The Supplemental Requirement for tensile tests in ASTM B 367 for Titanium and Titanium Alloy Castings designates 6.4 mm diameter tensile specimens to be used [53]. This requirement was developed to reduce material use for testing due to the relatively high cost of titanium compared to other metals such as steels.

Given the large prior beta grain sizes (~ 1 mm) of the cast material, a tensile specimen size-effect study was also conducted to determine the effect of specimen size on the tensile properties (especially ductility) of the titanium castings. For the cast and HIPed condition of the graphite mold casting (GMC 1), an additional nine specimens with the 6.4 mm diameter were tested, along with twelve specimens with tensile diameters of 3.2 mm and ten specimens with tensile diameters of 12.8 mm. The specimen geometries were similar to that shown in Figure 13, only scaled in size according to the diameter, retaining the 4 to 1 gauge length / gauge diameter ratio. Specifically, the gage lengths for the 12.5 mm, 6.4 mm, and 3.2 mm diameter specimens were 50 mm, 25 mm, and 6.4 mm, respectively.
3.3.1.3 Fracture Toughness

Fracture toughness testing was used to measure the energy required to grow a pre-existing sharp crack in a material. In this study, an elastic-plastic fracture mechanics approach was taken due to the extent of yielding around the crack tip, which would invalidate the linear elastic fracture mechanics approach. The J-Integral test method was utilized in accordance with ASTM E 1820 [54]. The “unloading compliance” test method was used to calculate J and develop a J-R curve for each casting condition. This technique was chosen because it utilizes only a single specimen to develop the J-R curve. In the test, the specimen undergoes multiple load/unload sequences where crack length is computed during each unload sequence based on the compliance measurements. Figure 14 shows the load/unload curve generated during the test. As the crack grows, the specimen becomes less stiff (more compliant), and thus, the crack length, and therefore, J can be calculated. This load/unload sequence is repeated until the specimen either fails.
by fracture instability or enough data are collected (load versus crack extension) to calculate a resistance curve for analysis using methods prescribed in ASTM E 1820.

The objective of the test is to load the fatigue pre-cracked specimen to induce either or both of the following responses: (1) unstable crack extension, referred to as fracture instability, and/or (2) stable crack extension. For each casting condition, and given the limitations in material available, two standard 25 mm thick compact tension specimens (Figure 15) were initially tested, and all tests were conducted at –2 °C. Tests were conducted at this temperature because -2 °C represents the lowest seawater temperature, and data at this temperature will allow for comparisons of cast Ti-5111 behavior to that of other alloys used by the Navy. The specimens were loaded in a servo-hydraulic test frame under actuator displacement control. They were fatigue pre-cracked to develop a sharp, pre-existing crack in the material of approximately 2.5 mm in length. Pre-cracking was conducted in air at ambient laboratory temperatures and at a frequency of 10 Hz. Subsequently, the specimens were side-grooved (10% of net thickness) to promote in-plane cracking during testing. If the specimen is not side-grooved, crack tunneling often occurs or shear lips could form due to the lower stress triaxiality state at the outer surfaces of the specimen.
Figure 14: Load vs. crack opening displacement (COD) curve generated using the “unloading compliance” method for measuring crack growth

Figure 15: Fracture toughness specimen geometry
To measure specimen compliance, a clip-gage was attached to knife edges mounted onto the specimen, as shown in Figure 16. The clip-gage consists of four strain gages mounted onto two cantilever beams. As the crack grows, the beams deflect causing a change in voltage across the strain gages, and thus, allowing displacement to be measured. An additional measurement of final crack growth was obtained after the test by heat-tinting the specimen at 315 °C for 30 minutes to mark the extent of crack growth. The specimen was subsequently removed from the furnace, air cooled, and fractured so that the final crack length could be physically measured.

For three selected cast conditions (GMC 1, GMC 4, and GMC 6), additional specimens were fabricated for a second lot of heat-treated plates, designated 2GMC 1, 2GMC 4, and 2GMC 6. An additional three specimens from each of these castings were tested using the same procedures described above.

In order to interpret the load-crack extension data, conventional elastic-plastic fracture mechanics analysis according to ASTM E 1820 was used. In this procedure, the J-integral is obtained and used to determine the onset of stable crack growth (\( J_{IC} \)). The J-integral measures the energy release rate as the crack grows in a nonlinear elastic body. The relationship between linear-elastic, nonlinear elastic, and elastic-plastic stress strain behavior is illustrated in Figure 17. The loading behavior for both nonlinear elastic material and elastic-plastic materials is identical; however, during the unload of an elastic-plastic material the specimen unloads linearly with a slope equal to the Young’s modulus of the material. A non-linear elastic material unloads along the path it was loaded. Thus, as Rice demonstrated
the assumption of nonlinear elastic behavior at the crack tip of metals with elastic-plastic behavior is valid assuming no unloading occurs.

The J-integral is also a way to determine the stress and strain conditions at a crack tip for materials that exhibit small-scale yielding. As long as the plastically yielded region at the crack tip is small compared to the specimen dimensions, and the specimen boundaries are remote from the crack tip and plastic zone, then the J-integral approach is a reliable measurement of fracture toughness for ductile materials [54,56]. Although the plastic region ahead of a crack can be significantly larger in a material characterized by \( J_{IC} \) rather than \( K_{IC} \), the size of the yielded region ahead of a crack tip must remain relatively small. To ensure a valid \( J_{IC} \) value is obtained, both the specimen thickness \( (B) \) and the remaining ligament length \( (W-a) \), where \( (a) \) is the crack length, must be considered. The specific specimen geometry and crack length/remaining ligament length requirements for obtaining a valid \( J_{IC} \) are given in Equation 1:

\[
B, b_0 \geq \frac{25J_Q}{\sigma_y}
\]  

(1)

where \( \sigma_y = 0.2 \% \) offset yield stress (kPa). Additionally, to maintain the assumption of non-linear elastic material behavior for a growing crack, the crack length must be updated throughout the test with new \( J_Q \) values computed based on the current crack length and remaining ligament length.
Figure 16: Instrumentation for measuring crack mouth opening displacement using a clip-gage [56]

Figure 17: Stress-strain behavior of a linear-elastic, non-linear elastic, and elastic-plastic material (schematic modified from Anderson [56])
The onset of stable crack growth is determined from the J-R curve, an example of which is illustrated in Figure 18 [56]. The J value for the material can be computed by separating J into its elastic and plastic components, Equations 2-4. J-elastic \((J_{el})\) is computed from the elastic stress intensity, \(K\), which is inferred from the load and crack size. The plastic component of J can be calculated from the area under the load-displacement curve generated during testing.

\[
J = J_{el} + J_{pl}
\]  
\[
J_{el} = \frac{K^2(1 - \nu^2)}{E}
\]  
\[
J_{pl} = \frac{\eta A_{pl}}{B_N b_0}
\]

where:

\(K\) = elastic plane strain toughness \((kJ/m^2)\),
\(\nu\) = Poisson’s ratio \((0.3\) for titanium),
\(E\) = elastic modulus \((117\ GPa\) for cast Ti-5111),
\(\eta\) = dimensionless constant = 2.26 for our specimen geometry,
\(A_{pl}\) = area under load-displacement curve,
\(B_N\) = specimen thickness \((mm)\),
\(b_o\) = ligament length of un-cracked material \((mm)\).

A provisional fracture toughness value, \(J_Q\), is computed from the J-R curve in Figure 18. As shown on the curve, exclusion lines are drawn at crack growths of
0.15 mm and 1.5 mm. The slope of these lines accounts for the crack-tip blunting that occurs in the material before ductile crack extension occurs. The data falling between these exclusion lines are fit to a power law of the form:

\[ J = C_1 (\Delta a)^{C_2} \]  

(5)

where \( \Delta a \) is the change in crack length, and \( C_1 \) and \( C_2 \) are constants, as described by ASTM E 1820. The value of \( J_0 \) is defined at the intersection of the 0.2 mm exclusion line and the \( J \) calculated from Equation 4. \( J_0 \) is equal to \( J_{IC} \) if the conditions in Equation 1 are met.
The $J_{IC}$ data can be converted to the fracture toughness of a material ($K_{JIC}$) by the following equation:

$$K_{JIC} = \sqrt{\frac{J_{IC}^* E}{(1-\nu^2)}}$$  \hspace{2cm} (6)

Additionally, the tearing modulus ($T$) was calculated out to 2.5 mm of crack growth. The tearing modulus is the slope of the resistance curve measured between $J_0$ and a certain amount of crack growth. The tearing modulus is indicative of the relative stability of the crack growth, and is useful because it provides a fuller understanding of the crack growth behavior in the material than just $J_{IC}$. By
considering the entire J-R curve behavior, one can better predict the crack growth behavior in a ductile metal. For example, the larger the tearing modulus, the less likely it is the material will exhibit unstable crack propagation. The tearing modulus is calculated from the J-resistance (J-Δa) curve using the following equation:

$$T = \frac{dJ}{da} \cdot \frac{E}{\sigma_y^2}$$

where \( \frac{dJ}{da} \) = average slope of the J-Δa curve between crack initiation and 2.5 mm of crack growth.

### 3.3.2 Hot-Rolled Plate

The mechanical property evaluation for the hot-rolled and annealed Ti-5111 was similar to that for the castings, but on a much smaller scale. The mechanical properties generated are used to compare with the casting properties and to better understand how castings and hot-rolled plate differ in behavior.

Because the plate was rolled, the mechanical properties between the rolling or longitudinal (L) direction and the transverse (T) direction may be different due to the induction of texture in the material. Therefore, the mechanical testing for the rolled plate was conducted such that the properties in both the longitudinal and transverse directions were measured. Hardness measurements were made in the same manner as that for the castings. Tensile and fracture toughness testing was conducted using the same procedures and ASTM standards outlined in the previous section on castings, but with a few minor differences detailed in the sections below.
3.3.2.1 Tensiles

Two tensile specimens 12.5 mm in diameter, and with a 4 to 1 length to diameter ratio, were tested for each the longitudinal direction and the transverse direction. The specimens were tested at a strain rate of $10^{-3}$ /s and at the ambient laboratory temperatures.

3.3.2.2 Fracture Toughness

Once again, the fracture toughness specimens were tested using the same methods and specimen geometry as described for the castings. Two specimens for each the longitudinal (L-T) and transverse (T-L) orientations were tested at -2 °C. The specimen orientation designations of (L-T) and (T-L) describe the loading (first letter) and crack propagation (second letter) directions.

3.4 Fracture Surface Characterization

3.4.1 Castings

3.4.1.1 General Fracture Surface Examination

The fracture surfaces of tensile and fracture toughness specimens were examined using a scanning electron microscope (SEM). The specimens were ultrasonically cleaned in both acetone and alcohol prior to observation in the SEM. Differences in fracture surface morphology based on heat treatment condition were analyzed.
3.4.1.2 Stereo-sectioning Fractography

Several fractured test specimens were examined using stereo-sectioning fractography. The fracture surface is coated with a protective lacquer and then cross-sectioned perpendicular to the fracture surface. Next, the specimen is mounted in dissolvable cold resin (Technovit 3040) which is then surrounded by a thermoset epoxy (EPOMET). The specimen is ground, polished, and etched using the procedures outlined in Section 3.2. Optical micrographs of the cross-sectioned specimen are taken and then the sample is soaked in acetone to dissolve the mount and remove the fractured specimen. The specimen is subsequently placed in the SEM and tilted at such an angle that both the fracture surface and corresponding polished cross-section can be observed. Using this procedure, features observed on the fracture surface can be directly correlated to the microstructure below the fracture surface allowing one to better understand the role of microstructure on the fracture behavior of cast titanium.

3.4.1.3 Surface Roughness Quantification

The surface roughness of the fracture toughness specimens was quantified using confocal microscopy. This technique was chosen over other surface roughness measurement techniques such as laser triangulation or profilometry because confocal microscopy was the only technique able to quantify the roughness of a sample with a large (~ 3 mm) range in surface roughness. The surface roughness (z-axis) resolution of confocal microscopy is on the order of nanometers with a range of up to 24 mm. Additionally, the fracture surfaces of the fracture toughness specimens have numerous steep angles, which also pose a significant
problem for most roughness measurement tools. Although confocal microscopy could still not define every point on the fracture surface due to shading by steep angles, it was still far better than alternative techniques and provided a reliable method to compare surface roughness of fracture toughness specimens from different castings conditions.

Confocal microscopy uses a specially designed optical microscope that measures vertical and lateral dimensions on a specimen, providing surface roughness information [57,58]. As illustrated in Figure 19, this microscope works by directing a thin beam of white light onto the surface of a specimen and imaging only the areas in focus. This is achieved through two methods: the illumination of a single point on the specimen means areas above or below the plane of focus quickly lose illumination, and a pinhole aperture is used to collect light reflecting back from the sample area in focus. Figure 19 is a schematic illustration of the microscope principles. Accurate height measurements on the specimen can be obtained by taking a series of thin optical sections on the z-axis. From these data, a three-dimensional topographic map can be composed by knowing the exact x, y, and z coordinates of each pixel on the image.

Based on the results of the fracture toughness testing, selected specimens were chosen to measure their surface roughness using confocal microscopy. The experiments were conducted on a Micromereasure – Nanovea Series microscope with a Micromereasure – CHR 150 High Resolution Optical Sensor spectrometer. The data were analyzed using MountainsMap software. For specimen GMC 1, a region approximately 10 mm x 5 mm was scanned, and for specimen GMC 7, a region 15
mm x 10 mm was scanned. These regions included data from the machined notch, fatigue pre-crack, crack-growth initiation region, and final fracture to provide an overview of the surface roughness between test regions. A step size of 40 μm across the thickness of the specimen and 10 μm vertically through the test region was used during the scans. This step size allowed for the large region to be measured in a timely fashion while still maintaining good resolution. Additionally, an area 25 mm x 3 mm in size was measured for GMC 1, GMC 3, GMC 7, IC 1, and IC 2 using the same step sizes. One fracture toughness specimen per condition was measured, except for GMC 1 and GMC 7 where two fracture toughness specimens were measured. The measured area consisted only of the crack-growth initiation region, used to determine J_{IC} and the tearing modulus, to determine the overall surface roughness of the crack growth region.
3.4.2 Hot-rolled Plate

The fracture surfaces of the tensile and fracture toughness specimens were studied using the SEM in the same manner as for the fractured casting specimens. Stereo-sectioning fractography and surface roughness measurements were not carried out on the hot-rolled plate.
Chapter 4: Quantitative Analysis of Ti-5111 Microstructures

4.1 Microstructure Development in Cast Ti-5111

The microstructure development in the as-cast and hot isostatically pressed (HIPed) plate is the result of a nucleation and growth process. As the casting cools below the beta transus (980 °C), the alpha phase begins to preferentially form at the beta grain boundaries, forming a mostly continuous layer of alpha along the prior beta grain boundaries. Subsequently, during slow to moderate cooling rates alpha laths (or plates depending on the thickness) nucleate either from the grain boundary alpha phase or from the prior beta grain boundary itself and grow into the beta matrix [14,60]. As the alpha laths grow, the beta soluble elements Mo, Fe, and V diffuse away from the migrating interface of the alpha laths and stabilize thin layers of retained beta phase between the alpha laths [61]. As the laths grow into the beta matrix, they grow parallel to one another forming alpha colonies. The parallel laths continue to grow until they intersect another alpha colony or prior beta grain boundary. Each of the alpha laths in a colony belongs to the same variant of the Burgers relationship for the alpha and beta phases. The Burgers relationship 

$(0001)_\alpha \parallel (110)_\beta$ and $[11-20]_\alpha \parallel [1-11]_\beta$ is maintained for each alpha lath or colony that develops.

As the cooling rate increases, the nucleation of new alpha colonies or laths begins to occur on other individual colonies or laths. These new alpha laths nucleate on the broad face of the existing alpha colonies and grow nearly perpendicular to them (typically at 80°) to minimize the overall elastic strains [14,60].
This specific angle is formed because the colonies will grow such that their basal poles are parallel, while their <11-20> poles are rotated by 10° from one another [60]. As the cooling rate continues to increase, the size of the alpha colonies decreases until eventually a basketweave structure is developed. The basketweave structure is comprised of individual alpha laths or small groups of alpha laths typically oriented approximately 80° to one another.

The microstructural development in the cast titanium alloy Ti-5111 was studied through a range of ten different heat treatment conditions of both the investment cast material and the graphite mold castings, as described in Table 3 of Chapter 3. Each beta anneal was conducted at 1010 °C, approximately 30 °C above the beta transus temperature. The alpha/beta anneals were conducted at either 843 °C (the standard annealing temperature for cast Ti-6Al-4V) or 954 °C (the standard alpha/beta annealing temperature for wrought Ti-5111). For each annealing condition, a cooling rate of 1 °C/min (corresponding to an industry furnace cool), 14 °C/min (corresponding to an industry air cool), or 55 °C/min (corresponding to an industry fan cool) was utilized. The different cooling rates enable an examination of the effect of cooling rate on the following: alpha lath width, alpha colony size, grain boundary alpha thickness, and volume fraction of alpha phase. The beta anneals are used to examine the effect of prior beta grain size on mechanical properties. Beta heat treatments typically increase the prior beta grain size due to grain growth at temperatures above the beta transus.

The microstructure developed in the graphite mold cast and HIPed specimen (GMC 1) is shown in Figure 20. The as-cast and HIPed specimen was cooled at
relatively slowly at a rate of 2 °C/min after the HIPing cycle. The sample developed an alpha colony structure with thin alpha laths and a thin, continuous layer of grain boundary alpha phase. Several of the alpha colonies are large (approximately 300 µm) and fill a significant portion of the prior beta grain. The development of the alpha and beta phases was verified by X-ray diffraction, and the results are provided in Appendix A.

The microstructure developed in the as-cast material (GMC 1) is useful as a baseline comparison with the microstructures that the graphite mold castings develop after the additional heat treatments detailed in Table 3. As shown in Figure 21, all of the graphite mold cast specimens exhibited a Widmanstatten alpha colony structure with grain boundary alpha phase decorating the prior beta grain boundaries. The average prior beta grain size for the as-cast and HIPed specimen was 1200 µm. As Table 4 shows, each specimen receiving a beta anneal has an average prior beta grain size smaller than the starting as-cast structure. The smaller grain size is a result of the nucleation of new, smaller grains during the beta anneal, as shown in Figure 23. These new grains likely nucleated from regions of residual stress developed from the collapse of porosity during the HIPing operation in the original casting. The specimens that did not receive a beta anneal (GMC 6 and GMC 7) could not nucleate new beta grains; and thus, have average prior beta grain sizes approximately equal to or slightly larger than the as-cast material.
Figure 20: GMC 1 – cast and HIPed at 900 °C, cooled at 2 °C/min
(a) GMC 2 – β anneal, cool at 14 °C/min

(b) GMC 3 – β anneal, cool at 1 °C/min
(c) GMC 4 – β anneal, cool at 14 °C/min, α/β anneal at 954 °C, cool at 14 °C/min

(d) GMC 5 – β anneal, cool at 1 °C/min, α/β anneal at 954 °C, cool at 1 °C/min
(e) GMC 6 – $\alpha/\beta$ anneal at 954 °C, cool at 14 °C/min

(f) GMC 7 – $\alpha/\beta$ anneal at 954 °C, cool at 1 °C/min

Figure 21: Optical micrographs of graphite mold cast Ti-5111 after heat treating
<table>
<thead>
<tr>
<th>Plate ID</th>
<th>Phase Region</th>
<th>Temp (°C)</th>
<th>Cooling Rate (°C/min)</th>
<th>*VFA</th>
<th>*PBGS (µm)</th>
<th>*ACS (µm)</th>
<th>*ALT (µm)</th>
<th>*GBAT (µm)</th>
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</thead>
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<tr>
<td>GMC 1</td>
<td>cast+HIP</td>
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<td>2</td>
<td>0.69</td>
<td>1200</td>
<td>180</td>
<td>3.0</td>
<td>7.4</td>
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<td>β</td>
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<td>14</td>
<td>0.62</td>
<td>1120</td>
<td>135</td>
<td>1.9</td>
<td>4.0</td>
</tr>
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<td>4.3</td>
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<td>β/α/β</td>
<td>1010 954</td>
<td>14</td>
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<td>920</td>
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<td>3.1</td>
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</tr>
<tr>
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<td>1 1</td>
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<td>1090</td>
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</table>

*VFA = volume fraction alpha
*PBGS = prior beta grain size
*ACS = alpha colony size
*ALT = alpha lath thickness
*GBAT = grain boundary alpha thickness
The data presented in Table 4 for alpha colony sizes are the average values for each microstructure feature. However, there is a large range of alpha colony sizes for each casting. As an example, a histogram quantifying the distribution of alpha colony sizes for casting GMC 4 is shown in Error! Reference source not found.. The highest frequency of data is observed between 40 – 50 µm, while the average colony size is 120 µm. The long tail at large colony sizes indicates that a considerable number of alpha colonies are much larger than the average. An analysis was conducted to compare the largest 5% of the alpha colonies of the various heat-treat conditions with the respective tensile and fracture toughness behavior (reported in Chapters 5 and 6). However, no significant correlation between the tensile/fracture behavior and the largest colony size was observed.

![Figure 22: Histogram showing the distribution of alpha colony size for specimen GMC 4](image-url)
The largest difference in microstructure of the heat-treated specimens was observed as a function of cooling rate. As shown in Figure 21 and Figure 23 and quantified in Table 4, the specimens cooled slowly at a rate of 1 °C/min exhibited significantly larger alpha colonies, coarser alpha laths, and thicker grain boundary alpha phase. For example, specimens GMC 2 (Figure 21a) and GMC 3 (Figure 21b) were both β-annealed at 1010 °C and cooled at two different rates. Specimen GMC 2, cooled at 14 °C/min exhibits a fine lath structure with an alpha lath thickness of 1.9 µm and a grain boundary alpha thickness of 4.0 µm, while the specimen cooled at 1 °C/min (GMC 3) exhibits a rather coarse structure with an alpha lath thickness of 4.3 µm and a grain boundary alpha thickness of 10.3 µm. Additionally, the alpha colony size is 135 µm for GMC 2 (14 °C/min) and 195 µm for GMC 3 (1 °C/min). The slower cooling rate allowed for longer diffusion times resulting in a growth of the alpha colonies, alpha laths, and grain boundary alpha phase.

Similar cooling rate differences can be noted between the specimens given a duplex anneal, which involves an additional α/β anneal (GMC 4, Figure 21c, and GMC 5, Figure 21d). Table 4 indicates the average lath thickness for the casting given an air cool (GMC 4) is 3.1 µm while that for the furnace cooled specimen is 5.1 µm. Following this trend, the grain boundary alpha thickness is 5.3 µm and 14.1 µm for GMC 4 and GMC 5, respectively. When compared to specimens receiving only the β anneal, the effect of a duplex anneal is thus evident through the thicker alpha laths and grain boundary alpha for each specimen given a β anneal followed by an α/β anneal. The α/β anneal at 954 °C resulted in more extensive diffusion and subsequent growth of the alpha laths and grain boundary alpha phase.
As expected, specimen GMC 6, receiving only an $\alpha/\beta$ anneal followed by a cooling rate of 14 °C/min (air cool), developed an alpha lath thickness (ALT) of 2.8 µm, a thickness in between that of the specimen given only a $\beta$ anneal and air cooled (ALT = 1.9 µm) and that given a duplex anneal and air cooled (ALT = 3.1 µm). The grain boundary alpha thickness (7.1 µm) was approximately the same as the as-cast condition, but larger than the specimen given a duplex anneal followed by an air cool (GMC 4).

The effect of cooling rate is not evident in the specimens receiving only an $\alpha/\beta$ anneal. The micrograph of specimen GMC 7 (Figure 21f) shows a microstructure very similar to its faster-cooled counterpart, specimen GMC 6 (Figure 21e). Table 4 shows quantitatively the similarities in the alpha lath thickness and grain boundary alpha thickness. It is possible that at the lower temperature of 954 °C, the rate of diffusion was decreased such that the cooling rate and time for diffusion did not play as large a role for these specimens. However, further investigations are necessary to completely understand the lack of microstructure coarsening in specimen GMC 7.

The volume fraction of alpha phase follows a similar pattern as the alpha lath thickness and grain boundary alpha thickness. The volume fraction of alpha phase (and correspondingly the volume fraction of retained beta) for the as-cast specimen GMC 1 is a function of both the alloy chemistry and the cooling rates from the initial casting process and from the HIPing operation. The volume fraction of alpha in the as-cast and HIPed plate is 0.69. The change in the volume fraction of alpha phase in the annealed specimens is a function of the annealing temperature(s), number of annealing cycles (single anneal or duplex anneal), and the cooling rate from each
anneal. The specimen given only a β anneal followed by an air cool (specimen GMC 2) developed the least amount of alpha phase ($V_f = 0.62$). This reduction in the volume fraction of alpha phase over the as-cast and HIPed specimen is due to the higher cooling rate from the beta phase field. Once the material is heated above the beta transus temperature, it transforms to all beta phase. Thus, the cooling rate from this temperature determines the amount of retained beta phase and resulting alpha phase at room temperature. Accordingly, the second beta annealed specimen (GMC 3) was cooled more slowly than the as-cast and HIPed specimen; and therefore, has a slightly higher volume fraction of alpha phase ($V_f = 0.74$).

Specimens GMC 4 and GMC 5 have the largest volume fraction of alpha phase because they both received duplex anneals, allowing greater time for diffusion to occur to stabilize the alpha phase. Within this group of specimens, the slower cooled specimen (GMC 5) developed slightly more alpha phase than the specimen cooled more quickly. Finally, the two specimens given only an α/β anneal (GMC 6 and GMC 7) have moderate volume fractions of alpha phase. Although these specimens did not receive a β anneal to transform their structures, they were heated high enough in the α/β phase field to allow further diffusion to occur and stabilize more alpha phase. Similar to the observations made for the alpha lath thickness, specimen GMC 7, which was cooled at a rate of 1 °C/min, did not develop more alpha phase than its faster cooled counterpart. Again, further investigations are needed to determine the cause of this behavior.

In summary, the microstructures developed from a single β anneal, a duplex anneal of β followed by an α/β anneal, and a single α/β anneal followed by a furnace
cool or air cool differed mainly due to cooling rate effects. The samples cooled slowly developed larger alpha colonies, thicker alpha laths, thicker grain boundary alpha, and a higher volume fraction of alpha phase. The as-cast and HIPed specimen developed a lamellar microstructure consistent with its moderate cooling rate from the HIPing operation. The specimens given a β anneal or duplex anneal followed by an air cool developed finer microstructures than the as-cast condition. The specimens undergoing these anneals, but followed by a furnace cool, developed significantly coarser structures than the as-cast condition. Finally, the two specimens given only an α/β anneal developed almost identical structures which were very similar to the as-cast condition. It was expected that the specimen receiving a furnace cool would have developed a coarser microstructure than it actually did.
Figure 23: Optical micrograph showing region of small prior beta grains nucleated during the $\beta$ anneals
The microstructure development in the Ti-5111 investment castings is similar to that in the graphite mold castings. As the micrographs in Figure 24 show, the castings developed a lamellar microstructure comprised of parallel alpha laths and grain boundary alpha phase outlining the prior beta grains. The most significant difference between the investment castings and the graphite mold castings is the larger prior beta grain size in the investment castings. The average prior beta grain size is approximately 1800 µm, while that for the graphite mold castings is 1200 µm. The larger prior beta grain size is due to the investment casting spending a longer period of time at elevated temperatures in the β phase field. This extended time in the β phase field can be attributed to a slower cooling rate of the original molten metal in the investment casting due to either the ceramic shell or a higher mold pre-heat. Unfortunately, detailed records of the casting process are unavailable.

Another noteworthy difference in the prior beta grain size in the two types of castings is the lack of recrystallized grains in the investment castings given a beta anneal. As noted in the graphite mold castings, local recrystallization occurred in the samples given a beta anneal, thereby reducing the average prior beta grain size. However, in the investment castings, both specimens given a beta anneal (IC 1 and IC 2) have average prior beta grain sizes only 50 µm smaller than the specimen that did not receive a beta anneal (IC 3). Additionally, metallographic examination showed no evidence of recrystallized grains in the investment castings.

Aside from the difference in prior beta grain size, the alpha lath thickness, alpha colony size, and grain boundary alpha thickness in the investment castings show the same trends as the graphite mold castings. The alpha colony size (ACS)
appears to be controlled more by cooling rate than by prior beta grain size. As Table 4 shows, the alpha colony size of the investment cast specimens is similar to that of the graphite mold casting for the same cooling rate. For example, specimen IC 3 and GMC 7 were both given alpha/beta anneals followed by a cooling rate of 1 °C/min. Both specimens developed comparable alpha colony sizes of 215 µm and 220 µm for IC 3 and GMC 7, respectively, even though the prior beta grain size of IC 3 was approximately 450 µm larger than GMC 7. Similarly, the alpha colony size of IC 1 was the smallest of all the cast specimens (ACS = 70 µm) and it received the fastest cooling rate of 55 °C/min. Specimen IC 2, receiving a duplex anneal followed first by a cooling rate of 55 °C/min from the beta phase field and then by a cooling rate of 14 °C/min from the alpha/beta phase field, developed an alpha colony size of 120 µm, the same as the graphite mold specimen receiving the duplex anneal followed by cooling rates of 14 °C/min.

The cooling rate in specimens IC 1 and IC 2 after the beta anneal (55 °C/min) was fast enough to develop a basketweave alpha structure in parts of the casting. Figure 25 shows a region of basketweave alpha in specimen IC 1. The fast cooling rate and large prior beta grains promoted the nucleation of alpha laths away from grain boundaries. Instead of nucleating at grain boundaries, these individual alpha laths and small packets of laths nucleated from other alpha laths with preferred orientation relationships.

The alpha lath thickness and grain boundary alpha thickness for the investment casting were controlled by the cooling rate, similar to the graphite mold cast specimens. Specimen IC 1 developed the finest structure with an alpha lath
thickness of 0.5 µm and a grain boundary alpha thickness of 2.5 µm. This sample had the fastest cooling rate of any of the investment or graphite mold castings; and consequently, developed the finest microstructure features. The microstructure of specimen IC 2 was slightly coarser than IC 1 (ALT = 2.0 and GBAT = 4.8) due to its additional anneal in the α/β regime followed by an air cool. Finally, specimen IC 3 (slow cooled at 1 °C/min after only an α/β anneal) developed the coarsest structure of the investment castings (ALT = 4.0 and GBAT = 5.6) after an α/β anneal followed by a furnace cool.

The volume fraction of alpha was also a function of cooling rate, as expected. The specimen with the fastest cooling rate (55 °C/min) retained the most beta phase with the volume fraction of alpha only 0.56. With slower cooling rates, specimens IC 2 and IC 3 had volume fractions of alpha phase of 0.74 and 0.84, respectively. The fast cooling rate retains more beta phase due to the lack of time for diffusion to occur. As the cooling rate decreases, more diffusion takes place stabilizing the alpha phase at room temperature.
(a) IC 1 – $\beta$ anneal, cool at 55 °C/min

(b) IC 2 - $\beta$ anneal, cool at 55 °C/min, $\alpha/\beta$ anneal at 954 °C, cool at 14 °C/min
(c) IC 3 – α/β anneal at 843 °C, cool at 1 °C/min

Figure 24: Optical micrographs of investment cast Ti-5111 after heat treating

Figure 25: IC 1 exhibiting basket weave alpha
4.1.2 Compositional Analysis

A relatively common problem with cast metal components is chemical segregation in the center of the material, particularly in thicker cross sections [47,62]. To ensure the graphite mold and investment castings were chemically homogeneous through the thickness, two specimens from each casting were analyzed for the weight percent of the elements Al, V, Mo, Sn, Zr, Si, Fe, and Ti through the thickness of each plate. The composition for specimens GMC 1, GMC 2, IC 1, and IC 2 were each measured, and representative results are illustrated in Figure 26. As the graph shows, the composition through the thickness of specimen GMC 1 was homogeneous; no segregation was observed on the scale of these measurements. Each of the other three specimens showed very similar results; therefore, only the data for GMC 1 are shown. It is noted that the vanadium level is approximately 0.5 wt % in Figure 27, which is significantly lower than the material certification sheets from Wah Chang (producers of the graphite mold casting) and PCC Structuralis (producers of the investment casting) who report an average vanadium weight percent of 1.02 and 1.01, respectively. Therefore, it is believed that the low weight percent value for vanadium is due to overlap between the vanadium and titanium X-ray lines. Because the titanium concentration is much greater than the vanadium concentration, residual overlap will cause the vanadium concentration to appear lower than actuality.

As discussed in Chapter 2, the elements Mo, V, and Fe are known to stabilize the beta phase, while Al stabilizes the alpha phase [4,13,18]. The neutral elements of Sn and Zr are considered equally soluble in both the alpha and beta phases.
[13,63]. However, the exact composition of the alpha and beta phase in most alloys has not been determined, with only limited data existing for the common Ti-6Al-4V alloy [64]. Therefore, the weight percent of the elements Al, V, Mo, Fe, Zr, and Sn was measured for the alpha and beta phases in the as-cast and HIPed graphite mold specimen (GMC1) with the results shown in Figure 27. The results for the as-cast condition (GMC 1) quantify the level of the partitioning for each element in the alpha and beta phases of Ti-5111. The figure indicates molybdenum has the greatest degree of solute partitioning with almost three weight percent in the beta phase and only 0.3 weight percent in the alpha phase. Vanadium and iron also have strong preferences for the beta phase. Aluminum does not exhibit as strong of a preference between the alpha and beta phases as the beta stabilizing elements of Mo, V, and Fe. Although aluminum segregates to the alpha phase (5.2 wt %), it is also present in the beta phase (3.8 wt %). Tin and zirconium are considered neutral elements with equivalent solubilities in the alpha and beta phases; however, in Ti-5111 both elements have a slightly higher presence in the beta phase.
Figure 26: Compositional analysis through the thickness of graphite mold cast Ti-5111 (specimen GMC 1) using electron probe X-ray microanalysis (EPMA)
Figure 27: EPMA analysis of the composition of the alpha and beta phases in the as-cast graphite mold specimen (GMC 1)
4.2 Microstructure Development in Hot-rolled and Annealed Ti-5111

The difference in mechanical property behavior, specifically tensile strength and ductility, as well as fracture toughness, between castings and hot-rolled and annealed plate is a function of their microstructures. The previous section provided a basis for understanding the microstructure development in cast Ti-5111 and its relationship to the heat treatment and cooling rates. To better understand the difference in strength, ductility, and fracture toughness between cast and hot-rolled Ti-5111, the differences in microstructure is considered below.

As described previously, the hot-rolled and annealed plate examined in this study was rolled in the beta phase field, air-cooled to room temperature, and heat treated in the alpha/beta phase field at 954 °C followed by air-cooling to room temperature. This processing procedure caused the rolled plate to develop a microstructure significantly different than its cast counterpart. The hot-rolled plate did not form a typical alpha colony or lamellar structure like the castings. Therefore, prior beta grains are not apparent, alpha colonies do not typically exist, and grain boundary alpha is either broken-up or no longer exists. Thus, several major microstructural characteristics of cast plate are not present in the hot-rolled and annealed plate.

Prior to hot working, the initial ingot microstructure is almost identical to the as-cast and HIPed graphite mold microstructure (GMC 1). The ingot is then heated into the beta phase region and rolled to 25 mm thick. Rolling in the beta phase field deforms the prior beta grains into non-recrystallized pancake-shaped grains. As the plate cools below the beta transus, grain boundary alpha phase forms along the
pancake-shaped grains, but it is discontinuous due to the deformation. Additionally, as the plate cools into the alpha/beta phase region, alpha plates nucleate from the deformed beta grains and grow inside the grain [46]. Occasionally, these alpha plates grow in small packets of parallel plates, but often they grow as individual plates. The time spent in the beta phase field and alpha/beta phase field during cooling is sufficient to promote coarsening of the alpha plates. Upon heating into the alpha/beta phase field for annealing, fine, secondary alpha laths develop in a transformed beta matrix surrounding the alpha plates [65].

Although processing in the beta region followed by an alpha/beta anneal is not common practice, several studies have been conducted on titanium processed in this manner. A similar microstructure was developed in the near-alpha titanium alloy Ti-6Al-2Sn-4Zr-6Mo after forging in the beta phase field and subsequently annealing 30 °C below the beta transus [46]. Ma et al [19] also described in detail the microstructure development of a near-alpha titanium alloy (Ti-6Al-2.7Sn-4Zr-0.4Mo-0.45Si) deformed in the beta phase region. This processing route was shown in both studies to produce a microstructure with relatively thick alpha plates in a fine transformed beta matrix.

The microstructure in the hot-rolled Ti-5111 plate is directional as a result of the rolling process. Therefore, a schematic of the plate directions is shown in Figure 28, and the corresponding microstructure from each direction is presented in Figure 29. As shown in the micrographs, the short and long transverse (TS and LS, respectively) directions show a coarse Widmanstatten microstructure consisting of thick alpha plates in a fine transformed beta matrix. The transformed beta matrix
consists of fine, acicular alpha laths and retained beta phase. The rolling (LT) direction of the plate shows the microstructure as it appears perpendicular to the transverse directions. Note the elongated grain structure in the TS and LS directions due to the rolling process. It is evident from these micrographs that the plate material did not recrystallize, but instead developed a deformed Widmanstatten structure. The significant deformation caused the grain boundary alpha phase to become discontinuous, the alpha colonies to become undefined, and the prior beta grains to become heavily deformed such that their boundaries are no longer well-defined.

The prominent microstructure features for the hot-rolled plate are the thickness of the alpha plates and the thickness of the acicular alpha laths within the transformed beta matrix. The microstructure of the hot-rolled plate shown in Figure 29b illustrates the thick alpha plates and fine acicular laths. The average alpha plate thickness measured from the transverse direction was 5.8 µm, while the average alpha lath thickness within the transformed beta matrix was 0.4 µm. The alpha plate thickness is larger than the alpha laths in any of the castings, while the alpha lath thickness in the transformed beta matrix of the hot-rolled plate is smaller than that of the castings.

These differences in microstructure between the Ti-5111 castings and hot-rolled and annealed plate are likely to affect the fracture behavior of the product types by changing the crack initiation and crack growth behavior in the material. The following chapters will consider in detail the influence of microstructure on the fracture behavior of the cast and wrought products.
Figure 28: Directions of the Ti-5111 hot-rolled plate
Figure 29: Microstructure of hot-rolled and annealed Ti-5111 plate showing (a) 3-D view of microstructure at low magnification and (b) micrograph of TS direction at higher magnification detailing the thick alpha plates and transformed beta matrix of fine alpha laths and retained beta
Chapter 5: Effects of Microstructure on Tensile Strength and Ductility

5.1 Tensile Properties of Cast Ti-5111

As stated previously, the Ti-5111 alloy was developed to have a minimum ultimate tensile strength of 689 MPa and a minimum yield strength of 586 MPa as described in the ASTM Standard B 265 for Alloy Grade 32 [53]. Additionally, the standard requires a minimum elongation of 10% for adequate ductility. Therefore, it is important to ensure that the tensile properties (both strength and ductility) remain above these minimum standards for the present heat-treated Ti-5111 castings. Several authors have suggested that the strength and ductility of wrought titanium alloys with lamellar structures is enhanced with a microstructure consisting of fine alpha laths and minimal grain boundary alpha phase [42,21,22,44,45]. However, none of these authors studied the influence of heat treatment and microstructure on cast titanium alloys. As discussed in Chapter 2, the differences in processing and resulting microstructure between wrought and cast titanium alloys leads to differences in their fracture behavior and mechanical properties.

For each heat treatment condition outlined in Table 3 of Chapter 3, two to four tensile specimens were fabricated and tested to obtain an initial comparison of the influence of heat treatment on the tensile strength and ductility of the Ti-5111 castings. A specimen diameter of 6.4 mm was utilized because the Supplemental Requirement for tensile tests in ASTM B 367 for Titanium and Titanium Alloy
Castings designates this specimen size to be used [66]. The number of specimens tested was limited by material availability.

The results for the initial set of tensile tests are provided in Table 5. As the data show, each of the castings exceeded the minimum ultimate tensile strength and yield strength requirements for this alloy. Significantly higher strength values were observed in the as-cast and HIPed condition of the graphite mold casting (GMC 1) and the beta annealed and fan-cooled investment casting (IC 1). The higher strength exhibited by casting GMC 1 is consistent with the higher hardness of the material; see Table 5. With the exception of GMC 1, each of the other graphite mold cast specimens showed similar strength and hardness values.

Among the investment cast specimens, the higher strength of investment cast specimen IC 1 can be attributed to its fine lath structure and basketweave alpha developed in the material as a result of the fast cooling rate (54 °C/min). It should be recalled that Table 4 showed the beta annealed and fan-cooled plate (IC 1) formed alpha laths of only 0.5 µm thick, four times smaller than the alpha lath thickness of any other casting condition. A fine alpha lath structure has been shown to reduce the slip length in the material, which in turn, enhances the strength [42,38,22]. Similarly, smaller alpha colonies decrease slip length in the casting and lead to enhanced strength [42,22,45,48]. Referring back to Table 4, IC1 formed the smallest alpha colonies with an average size of 70 µm, 60% smaller than any other casting. Additionally, a basketweave microstructure leads to higher hardness and higher strength than a lamellar microstructure [42,22,44].
Table 5: Tensile Properties of Cast Ti-5111 Plate

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<th>0.2% Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction of Area (%)</th>
<th>Hardness (HRC)</th>
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<td>10 minimum</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

* β = 1010 °C, α/β = 954 °C, FAC = fan air cool (54 °C/min), AC = air cool (14 °C/min), FC = furnace cool (1 °C/min)

** α/β = 843 °C
The ductility of the castings is characterized by a great deal of scatter in both the percent elongation and the percent reduction in area. Several of the specimens were below the Navy’s 10% minimum elongation benchmark for adequate ductility in shock applications. As Table 5 shows, a large variation in ductility values resulted both between castings and within a single casting set. In some cases, both elongation to failure and reduction of area values varied by a factor of two within the three specimen test set. Despite relatively similar strength levels, the elongation ranged from a low of 5% to a high of 13%, while the reduction-in-area values ranged from 11% to 26%. Additionally, the specimens with low elongation had reduction in area values only slightly greater. For example, specimen GMC 3-1 had an elongation of 7% and a reduction in area of only 11%.

The large scatter in elongation and reduction in area, as well as the low reduction-in-area values, are characteristic of fracture controlled by crack initiation and propagation [67,68]. In such cases, the strain to nucleate a crack, as well as the strain to propagate a crack, will vary based on the orientation and size of the microstructure feature that cracks. Thus, a large variation in ductility between specimens can be expected. On the contrary, fracture controlled by damage accumulation (void nucleation, growth, and coalescence) consistently allows for significant deformation below the fracture surface. Thus, the ductility values (both elongation and reduction in area) exhibit minimal scatter.

One method to determine what controls the fracture of these tensile specimens is to examine the fracture surfaces of the specimens. The fracture surfaces of two specimens from the same heat treatment (beta anneal at 1010 °C,
furnace cool, α/β anneal at 954 °C, furnace cool) with significant differences in ductility (GMC 3-2 (elongation = 13%) and GMC 3-1 (elongation = 7%)) were examined and are presented in Figure 30. The fracture surface of the high ductility specimen GMC 3-2 shows a much rougher surface and a fracture profile somewhat similar to the common cup-and-cone fracture characteristic of high ductility. Significant deformation on the outer surface of the specimen occurs and specimen necking results in a much larger reduction in area (27%).

In contrast, Figure 30b and Figure 31 show the presence of a large, relatively flat facet on the fracture surface of the low ductility specimen. The facet, which is outlined in Figure 30b and extends ~1800 μm along the specimen surface, suggests a microstructural feature on the scale of 1000-2000 μm deformed and cracked in a coordinated manner such as to create a large, relatively planar crack that subsequently propagated and limited ductility. The result is a small elongation to failure (7%) as well as a small reduction of area (11%). Thus, these results support the hypothesis that fracture of at least the low ductility specimens occurred by crack initiation and subsequent crack growth.

To relate the flat facet on the fracture surface of specimen GMC 3-1 to its corresponding microstructure, stereo sectioning and imaging was used [69]. The specimen was cross-sectioned perpendicular to the fracture surface, and subsequently ground, polished, and etched, as shown in Figure 32. This figure shows the large facet on the fracture surface directly corresponds to a large prior beta grain (and a single alpha colony) with a linear intercept of approximately 1500 μm. Importantly, Figure 32a indicates a large segment of the colony intersects the
specimen's outer surface. Furthermore, no voiding is evident beneath the fracture surface suggesting that this grain was oriented such that a crack was able to nucleate and propagate through the entire grain with minimal resistance. Cracks typically initiate more easily on specimen surfaces because a surface grain can deform predominantly by activating a single slip system, as opposed to interior grains that need 3-5 slip systems activated to deform due to grain boundary constraints. As a result, within large grains, large slip offsets / displacements will be created along slip bands intersecting the free surface, presumably promoting fracture along these bands. Thus, with regard to tensile ductility, these results indicate the importance of the presence of large beta grains and alpha colonies residing on the specimen surface where cracks can be most easily initiated.
Figure 30: Fracture surface of specimen (a) GMC 3-2 exhibiting 13% elongation and (b) specimen GMC 3-1 exhibiting low ductility (7% elongation)

Figure 31: Low-energy fracture of large facet from the low ductility specimen GMC 3-1
Figure 32: (a) stereo micrograph correlating facet on fracture surface with the underlying microstructure and (b) optical micrograph showing large prior β grain correlating to large facet from the low ductility specimen GMC 3-1.
The fracture surfaces, such as those shown in Figure 30, suggest the presence of a large microstructural feature on the scale of 1500-2000 μm can fracture and initiate a large crack that then significantly limits ductility by subsequent crack growth. Figure 33 shows two adjacent Widmanstatten colonies that share an orientation relationship allowing neighboring, planar slip bands to extend 2000 μm across the boundary between two large colonies with no apparent deviation. Importantly, several cracks are evident having formed along the intense, planar slip bands. Such long slip band distances are known to promote crack initiation [37], and in this case, the result is a planar fracture surface and comparatively low tensile ductility (elongation of this specimen was 8 percent). Thus, decreasing the prior beta grain size should improve ductility. Table 4 and Table 5 support such a relationship in that castings with the smallest prior beta grain size (i.e. GMC 4 and GMC 5) had the highest ductility.

The alpha colony size should also influence the tensile ductility by controlling the initial crack length developed under tensile loading. However, prior beta grain size is often more influential in determining the initial crack length and tensile ductility. Bridier et al. [70] have shown slip can transfer easily across alpha colonies with drastically different geometric orientations. They and others [48,49,70] showed that alpha colonies often have very similar crystallographic orientations even when their geometric orientations are seemingly quite different. Therefore, if alpha colonies have similar crystallographic orientations then the prior beta grain size will control slip length, and thus, crack initiation and propagation length. However, if the alpha colonies have significantly different crystallographic orientations, then the
alpha colony size will likely control the effective slip length and crack length in the material, and some previous studies have shown alpha colony size limits the slip length [22,45], and thus, tensile ductility of a lamellar titanium alloy.

Figure 33: The cross-section of as-cast tensile specimen with low 8% elongation (specimen GMC 1-3) showing slip bands parallel to planar fracture surface that extend across two large colonies and several smaller ones (optical micrograph utilizing polarized light)
Part of this study involved determining the microstructural condition that provides the optimum tensile strength, ductility, and fracture toughness combination. Thus, additional tensile testing was conducted on the following three graphite mold castings: GMC 1 (as-cast and HIPed), GMC 4 (beta anneal, air cool, alpha/beta anneal, air cool), and GMC 6 (alpha/beta anneal, air cool). These castings were chosen for additional testing based on the results of the initial tensile test data shown in Table 5, as well as initial fracture toughness test results (discussed in Chapter 6). For each of these castings, nine additional tensile specimens of 6.4 mm in diameter were tested.

As shown in Table 6, the as-cast and HIPed condition (GMC 1) had slightly higher average strengths than the GMC 4 and GMC 6 castings; see Table B-1 for individual specimen data. The ductility values for this group of 27 specimens are quite similar, and importantly, the scatter in the ductility values in Table 6 is significantly lower than that observed in the original group of GMC specimens tested (Table 5). Each casting condition in Table 6 showed little scatter within their datasets; specifically, the standard deviation for any casting group did not exceed 1.1 percent.

A possible reason for the reduced scatter in the tensile ductility for GMC 1, GMC 4, and GMC 6 is their relatively small alpha colony size. Because each of these specimens was air cooled from their respective annealing treatment, the alpha colony size remained relatively small. As presented in Table 4, the average colony sizes for these cast conditions are 180 µm, 120 µm, and 160 µm, respectively. It was observed in Figure 30 for GMC 3-1 that a large alpha colony / prior beta grain
fractured and limited the tensile ductility. By maintaining a relatively small alpha colony size, the crack size that can initiate remains small and does not drastically affect the tensile ductility.

Table 6: Additional Tensile Data for Select Casting Conditions
(GMC 1, GMC 4, and GMC 6)

<table>
<thead>
<tr>
<th>Casting</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>0.2% Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction of Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GMC 1 (as-cast &amp; HIPed)</td>
<td>Average St. Deviation</td>
<td>799</td>
<td>760</td>
<td>9.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5</td>
<td>5</td>
<td>1.1</td>
</tr>
<tr>
<td>GMC 4 *(β anneal, AC, α/β anneal, AC)</td>
<td>Average St. Deviation</td>
<td>792</td>
<td>748</td>
<td>10.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>6</td>
<td>1.1</td>
</tr>
<tr>
<td>GMC 6 *(α/β anneal, AC)</td>
<td>Average St. Deviation</td>
<td>782</td>
<td>741</td>
<td>9.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6</td>
<td>4</td>
<td>0.7</td>
</tr>
</tbody>
</table>

*AC = air cool (14 °C/min), β anneal @ 1010 °C, α/β anneal @ 954 °C
** averages based on 9 specimens for each condition
5.2 Tensile Specimen Size Effect Study

Tensile fracture controlled by a crack initiation and propagation process should have a greater influence on the tensile behavior of a small diameter specimen because a long crack of given length (such as a prior beta grain size) will be more detrimental to a specimen 3 mm in diameter than a specimen 12.5 mm in diameter. The microstructure features such as prior beta grain size (average = 900 µm – 1800 µm) and alpha colony size (average = 70 µm – 230 µm) likely control the length of the initial crack formed in the castings under tensile loading. In tensile specimens of only 3.2 mm in diameter, a crack of 1.8 mm in length will be more influential than a crack of similar length in a larger specimen. Therefore, this section explores the influence of the specimen size on the tensile behavior.

The casting condition chosen for this study was the graphite mold as-cast and HIPed material (GMC 1). Nine to twelve tensile specimens were tested with the following diameters: 3.2 mm, 6.4 mm and 12.5 mm. In all cases, the gage length to diameter ratio was maintained at 4:1.

As shown in Table 7 and Figure 34, increasing specimen diameter increases both the yield and tensile strengths in a manner that exceeds experimental scatter (see Table B-2 for data). Also apparent from the data is that a decrease in specimen size leads to an increase in standard deviation of strength values, from a standard deviation of 5 MPa in strength for the two larger specimens, to a standard deviation of 16 - 21 MPa for the smallest diameter specimens. Finally, Figure 34 shows little influence of specimen size on the average tensile ductility values.
although the standard deviation of ductility values increased significantly as the specimen size decreased.

Table 7: Tensile Properties as a Function of Specimen Diameter for Casting GMC 1 (As-Cast and HIPed)

<table>
<thead>
<tr>
<th>Specimen Size</th>
<th>Specimen No.</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>0.2% Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction of Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.5 mm dia.</td>
<td>Average St. Deviation</td>
<td>814 5</td>
<td>777 5</td>
<td>10.0</td>
<td>23.0 1.6</td>
</tr>
<tr>
<td>6.4 mm dia.</td>
<td>Average St. Deviation</td>
<td>799 5</td>
<td>760 5</td>
<td>9.9</td>
<td>26.0 2.7</td>
</tr>
<tr>
<td>3.2 mm dia.</td>
<td>Average St. Deviation</td>
<td>791 21</td>
<td>743 16</td>
<td>8.7</td>
<td>19.5 5.3</td>
</tr>
</tbody>
</table>
Figure 34: Graphical representation of the effect of specimen size on the strength and ductility of graphite mold cast Ti-5111 (GMC 1)
The relationship between specimen size and grain size can be visualized schematically in Figure 35. In the case of the small diameter specimens, the majority of the grains intersect the specimen surface. An approximation of the number of surface grains ($N_s$) on the 3.2 mm diameter specimen and the 12.5 mm diameter specimen can be made by calculating the specimen circumference and dividing by the average grain size, as given in Equation 8. A comparison between specimen sizes is calculated by obtaining a percentage of grains adjacent to the surface using Equations 9 and 10.

$$\# \text{ of surface grains (} N_s \text{)} = \frac{\text{specimen circumference}}{\text{ave. grain size}} \quad (8)$$

$$= \frac{\pi d}{1.2 \text{ mm}}$$

where $d = \text{specimen diameter}$

$$\# \text{ of total grains (} N_{\text{TOT}} \text{)} = \frac{A_{\text{specimen}}}{A_{\text{grain}}} \quad (9)$$

$$A_{\text{specimen}} = \pi r^2$$

$$A_{\text{grain}} = \frac{3}{2} r^2 \sqrt{3} \quad \text{(assuming hexagonal grain)}$$

where $r = \frac{\text{(grain size)}}{2} = 0.6 \text{ mm}$

The fraction of surface grains ($F_S$) is given by:

$$F_S = \frac{N_s}{N_{\text{TOT}}} \quad (10)$$

For a 3.2 mm diameter specimen, approximately 85% of the grains will be located adjacent to the specimen surface. In contrast, only approximately 25% of the grains are adjacent to the surface of a 12.5 mm diameter specimen.
Surface grains tend to be softer than interior grains because they can deform by slip along a single plane at stresses lower than those required to initiate multiple slip systems in interior grains, which are constrained by surrounding grains and need 3-5 independent slip systems activated to plastically deform. Thus, yield strength is decreased by specimen size because small specimens have a number of large grains located on the specimen surface (~85%), and these grains can subsequently yield at lower stress levels via single slip.
Figure 35: Schematic cross-section of (a) 3.2 mm diameter tensile specimen and (b) 12.5 mm diameter tensile specimen, both with grain sizes of approximately 1.2 mm.
Figure 34 shows that although there is little change in the average tensile ductility as a function of specimen size, the standard deviation for the tensile ductility gradually increases as the specimen size decreases. Table 7 shows the scatter within the elongation and reduction in area was quite large for the smallest specimen; the elongation ranged from a low of 4 percent to a high of 12 percent while the reduction in area ranged from a low of 11 percent to a high of 29 percent.

For the low ductility specimen (4% elongation to failure), Figure 36 shows an abrupt loss in load associated with the initiation of a large crack. In contrast, the high ductility specimen (12% elongation to failure) exhibits much more extension before a decrease in load-carrying capacity occurs near failure; no premature abrupt loss of load occurred in this case.

Examination of the fracture surface of the low ductility specimen GMC 1-33 (Figure 37a) shows that fracture occurred due to crack initiation and propagation forming two large, relatively smooth facets intersecting at the center of the specimen. The corresponding microstructure shown in Figure 38b indicates only two grains across the diameter of the specimen that share a similar orientation relationship allowing slip to transfer relatively easily across the grain boundary. As a result, intensive slip occurred in a concentrated planar manner through several colonies, initiating a large crack that limited ductility.
Figure 36: Tensile curves for the low and high ductility specimens GMC 1-33 and GMC 1-25, respectively.
In contrast, the high ductility specimen GMC 1-25 (12% elongation) exhibited a macroscopically rough fracture surface surrounding a single flat facet, as is shown in Figure 37b and Figure 38b. Examination of the microstructure below the flat facet in Figure 37b showed the facet corresponded to fracture along a slip plane through a single alpha colony. This specimen showed evidence of numerous active slip planes that allowed for significant deformation to occur before fracture. Figure 39a shows a high density of slip planes and considerable deformation of the beta phase.

In contrast, the low ductility specimen GMC 1-3 (previously shown in Figure 33) shows much fewer activated slip planes and minimal deformation of the beta phase beneath the fracture surface; see Figure 39b. Figure 39b also shows evidence of large shear offsets within the beta phase.

A combination of large alpha colonies and large prior beta grains lead to large slip offsets due to localized slip over large distances that create long cracks in the material. Thus, a decrease in the prior beta grain size and/or alpha colony size will limit the slip length in the material, and thus, limit the initiation of large cracks.
Figure 37: Fracture surface and corresponding microstructure of (a) the low ductility specimen GMC 1-33 showing a relatively smooth fracture surface and (b) the high ductility specimen GMC 1-25 showing a more tortuous fracture surface
Figure 38: Cross-sections contrasting the fracture profile of (a) the high ductility specimen GMC 1-25 with (b) the low ductility specimen GMC 1-33

(Optical micrographs taken under polarized light)
Figure 39: SEM micrographs showing the microstructure below the fracture surface of the high ductility (12% elongation) specimen GMC 1-25 showing extensive slip and deformation of the beta phase and (b) the low ductility (8% elongation) specimen GMC 1-3 showing fewer slip planes and large shear offsets along the slip lines
Occasionally, void formation and cracking at alpha colony boundaries was observed, as shown in Figure 40. Such damage behavior can be expected due to strain incompatibility between the alpha colonies, which depends on the individual crystallographic misorientation of the neighboring colonies. Thus, local regions of strain incompatibility will develop at some alpha colony boundaries leading to higher local stress states and void formation and cracking.

**Figure 40:** SEM micrograph showing void nucleation and crack growth at alpha colony boundaries in as-cast specimen GMC 1-25 (12% elongation)
In summary, tensile ductility and fracture in cast Ti-5111 is controlled by a crack initiation and propagation phenomenon, which is a result of the slip behavior in the material. In most instances, ductility is limited by the strain required to initiate a crack. Once a crack initiates (typically along slip planes through alpha colonies and across prior beta grains, and occasionally at alpha colony boundaries) the strain required to propagate the crack is relatively low. Whether the crack transverses across alpha colony boundaries and through an entire beta grain is dependent upon the crystallographic orientation of the alpha colonies. Cracks initiate most readily along intense slip planes that have large slip distances and therefore large slip offsets of the more ductile beta phase. The resulting cracks grow easily through large alpha colonies and prior beta grains; limiting the size of these features will improve the material’s ductility.

An engineering conclusion from the tensile testing of these 3.2 mm, 6.4 mm, and 12.5 mm diameter casting specimens is that a specimen size of 12.5 mm yields the most accurate representation of material behavior given a large number of grains across the specimen diameter. For testing purposes and quality assurance measurements, however, a specimen size of 6.4 mm in diameter provides results similar to that of its larger sized counterpart, but with less material cost. Testing of tensile specimens of cast titanium alloys only 3.2 mm in diameter should be limited to applications where the cross-sectional area of a component is approximately equivalent to that of the sub-size tensile specimen.
5.3 Comparison between Cast and Wrought Ti-5111 Tensile Properties

The previous sections have shown that tensile fracture in cast Ti-5111 is controlled by a crack initiation and propagation phenomenon associated with large prior beta grains and colonies within a cast material. In contrast, the microstructure features (prior beta grains and alpha colonies) that control slip length and crack initiation in the castings are not present in hot-rolled and annealed Ti-5111, compare Figure 41a and b.

In general, the ductility of the hot-rolled material was slightly greater than that of the castings, and better reproducibility was obtained. Each specimen tested achieved elongations of 12% or greater and reduction of area values greater than 23%. The relatively large differences in elongation and reduction-in-area values suggest fracture did not occur by a crack initiation and propagation process.

As shown in Figure 42, the fracture surfaces of the hot-rolled plate (exhibiting 13% elongation) exhibit several of the characteristic features of classic ductile fracture characterized by void nucleation, growth, and coalescence with a cup-and-cone fracture profile. The fracture surface is characterized by microvoids that likely formed at alpha/beta interfaces; see Figure 42b. Ductile tearing of the beta matrix is observed between the voids. As is typical of ductile cup-and-cone fracture surfaces, shear lips and shear dimpling was observed near the edge of the fracture surface as shown in Figure 42c. Thus, fracture surface features observed on the cast Ti-5111 tensile specimens, such as a tortuous fracture surface with numerous planar crack deviation planes are not observed in the hot-rolled plate.
The effective slip length in the hot-rolled plate is limited to the size of the individual alpha laths (< 10 µm); therefore, easy initiation of long cracks and subsequent propagation as observed in the castings is not likely to occur in the wrought plate. Additionally, no smooth facets were observed on the fracture surface of the wrought tensile specimens that would indicate crack initiation related to large microstructure features. Thus, the hot-rolled and annealed Ti-5111 exhibited somewhat higher ductility values over the cast Ti-5111. The enhanced ductility is due to fracture controlled by void nucleation, growth, and coalescence, as opposed to crack initiation and propagation.

Finally, as shown in Table 8, the transverse orientation of the hot-rolled plate exhibits higher tensile and yield strengths than the longitudinal orientation, as is typical in titanium alloys with an hcp crystal structure [13,71]. Hot-rolling above 900 °C, as was the case for the Ti-5111 plate, typically produces a weak texture in the plate such that the basal plane of the hcp crystal structure is oriented perpendicular to the rolling direction [71], as shown schematically in Figure 43. The strength in the transverse direction is greater due to the difficulty of prismatic slip in the material when stressed in the transverse direction [71]. Higher applied stresses are required to initiate slip and plastically deform the material.
Figure 41: Comparison of the (a) alpha colony microstructure developed in cast Ti-5111 and the (b) Widmanstatten alpha microstructure developed in hot-rolled and annealed Ti-5111
Figure 42: Fracture surface of hot-rolled Ti-5111 tensile specimen (longitudinal #2 – 13% elongation) showing (a) a macroscopically smooth fracture surface as compared to fractured cast Ti-5111 tensile specimens, (b) ductile void formation on surface, and (c) shear dimpling near outer edges of specimen.
Table 8: Tensile Properties of 1”-thick Hot-Rolled and Annealed Ti-5111 Plate (as determined from 12.5 mm-diameter specimens)

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>0.2% Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction of Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Longitudinal</td>
<td>835</td>
<td>729</td>
<td>12.0</td>
<td>23.6</td>
</tr>
<tr>
<td></td>
<td>833</td>
<td>726</td>
<td>13.0</td>
<td>25.1</td>
</tr>
<tr>
<td>Transverse</td>
<td>853</td>
<td>752</td>
<td>12.0</td>
<td>23.3</td>
</tr>
<tr>
<td></td>
<td>856</td>
<td>753</td>
<td>12.0</td>
<td>26.3</td>
</tr>
<tr>
<td>ASTM B 265, Grade 32</td>
<td>689 minimum</td>
<td>586 minimum</td>
<td>10 minimum</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 43: Schematic of hot-rolled plate with texture such that the basal plane of the hcp crystal is normal to the rolling direction (RD)
Chapter 6: Effect of Microstructure on the Fracture Toughness and Tearing Modulus of Cast Ti-5111

6.1 Fracture Toughness of Cast Titanium and ASTM Standard E1820

The origins of elastic-plastic fracture mechanics date back to 1968 when Hutchinson [72] and Rice and Rosengren [73] developed a singularity solution for stresses and strains near a planar, two-dimensional crack in a power hardening elastic-plastic material. Based on this, Rice [74] was able to define J as a path-independent variable. The initial materials used to develop and test the J-integral theory were steels, presumably because the J-integral test method was developed by persons from the Naval Research Laboratory, and thus, the primary interest was structural ship materials.

The majority of materials tested using the J-integral test method (defined in ASTM E 1820 [54]) have been wrought products where the grain size of the material has typically been 100 µm or less. Testing a casting poses certain issues due to its large grain size, such as that of the graphite mold castings (approximately 1.2 mm). When testing a standard 25 mm thick compact tension specimen for the graphite mold castings, there are only approximately 18 - 20 grains across the thickness of the specimen. Therefore, if crack growth across properly oriented grains is easy, the orientation of those grains will play a significant role in the overall crack propagation
behavior of the material. Specifically, if a grain or colony is oriented such that crack
growth can occur easily, then the fracture initiation toughness and crack propagation
resistance may be significantly decreased.

A related concern with ASTM E 1820 is that it requires the calculation of $J_{IC}$ to
be made at 0.2 mm of crack growth. Because the grain size and alpha colony size
is quite large in a casting and a crack can easily follow the length of the grain
boundary or alpha colony, only a few large grains or colonies would need to crack
for the compliance of the specimen to decrease such that it is perceived the entire
crack has grown 0.2 mm, when in fact only a few grains or colonies had fractured.
To quantify the number of grains needed to crack in the material to cause the
appearance of 0.2 mm of crack growth, an approximation of the crack growth area
was made for both a standard specimen with a straight crack front and that for a
specimen with a grain size of approximately 1.2 mm where only a few grains
cracked.

Figure 44a is a schematic of 0.2 mm of crack growth in a material with a
relatively smooth crack front. The area of crack growth is taken as the product of the
width of the test specimen (20.3 mm) and the length of crack growth (0.2 mm).
Thus, the cracked area of an ideal specimen measured using the ASTM E 1820
criteria is approximately 4.1 mm$^2$. To determine the number of grains that would
need to crack to achieve roughly 4.1 mm$^2$ of cracked material, and thus a material
compliance that would measure 0.2 mm of crack growth, the surface area of a grain
was assumed to be equal to that of a hexagon. The area (A) of a hexagon is given
by:
where “r” is the distance from the center to a corner. Thus, assuming a grain size of 1.2 mm, then \( r = 0.6 \) mm, and the area of a grain is equal to 0.94 mm\(^2\). Therefore, about 20% of the grains (approximately 4 to 5 grains) would need to crack completely to give the appearance of 0.2 mm of crack extension. Figure 44b shows a typical crack growth region where several grains or large colonies have fractured.

Although the calculation for the number of grains required to crack to cause an appearance of 0.2 mm of crack extension is a simplified example, it clearly suggests a role of properly oriented large grains in causing a decrease in the \( J_0 \) value as determined by the measurement requirements of ASTM E 1820.

In an attempt to overcome the limitations of ASTM E 1820 for large-grained materials, the fracture toughness of each casting was also measured at a crack length equal to the average grain size for that specific casting. The effects of this novel method of measuring fracture initiation toughness in castings will be considered in the following section.
Figure 44: Schematic fracture toughness specimens contrasting (a) an ideally straight crack front and (b) one that follows large microstructure features.
6.2 Fracture Toughness of Cast Ti-5111

The wrought Ti-5111 alloy was developed to have high fracture toughness (Navy goal of \(J_{IC} = 685 \text{ kJ/m}^2\)); however, differences in the fracture toughness of the cast product form due to differences in microstructure had not previously been considered. The fracture toughness for each heat treatment condition of the graphite mold castings and the investment castings was determined. A subsequent analysis of the toughness behavior and associated crack paths forms the basis for understanding the role of microstructure on the fracture toughness of the Ti-5111 alloy in the cast form.

For each casting condition, two to five specimens were tested in accordance with ASTM Standard E 1820 using the single-specimen J-integral technique, as described in detail in the Experimental Procedures Chapter. Each sample was fatigue pre-cracked and subsequently tested using the load/unload test technique to achieve 2.5 mm of stable tensile crack growth, the amount required to determine the tearing modulus of the material. One-half of a fracture toughness specimen (GMC 2-2) is shown in Figure 45. The boundaries between the fatigue pre-crack, tensile crack growth, and final fracture regions are shown. Physical measurements of the crack length for both the pre-crack and the tensile crack growth regions were obtained and compared to that determined by the J-test software during testing.
Figure 45: Fracture surface of fracture toughness specimen GMC 2-2 after heat tinting and fracturing to reveal fatigue pre-crack and tensile crack growth region
An example of the methodology used in analyzing the J-Δa crack growth behavior is shown in Figure 46. In this case, prior to the onset of crack growth, the specimen absorbed 90 kJ/m² of energy as blunting at the crack tip occurred. Once enough energy is absorbed at the crack tip, the crack begins to grow through the plastically deformed region ahead of the crack tip. According to ASTM E 1820, as crack growth occurs, it is monitored by the partial unloads, and the points shaded in black are used to determine a least squares fit to the J-Δa behavior. The intersection between the fitted line and the 0.2 mm offset line is taken as the fracture toughness. For this specimen, the fracture toughness is equal to 122 kJ/m².

As defined in Equation 7 of Chapter 3, the tearing modulus, T, is a measure of the material’s resistance to continued crack growth. The tearing modulus of each specimen was determined by calculating the slope of a linear-fit line between 0.15 mm and 2.5 mm of crack growth. For specimen GMC 4-4, shown in Figure 46, the tearing modulus equals 12.0.
Figure 46: J-Δa curve for specimen GMC 4-4 (β anneal at 1010 °C, air cool, α/β anneal at 954 °C, air cool)

The results of the fracture toughness determinations for each of the castings are shown in Table 9, and Table 10 is repeated from Chapter 4 to allow for easy comparison between the fracture toughness and microstructure features for each casting. In addition to $J_q$ values determined using the analysis procedure in ASTM E 1820 (where the fracture toughness is obtained at 0.2 mm of crack growth), Table 9 also includes a second set of $J_q$ values corresponding to a crack growth increment equal to the average grain size for each specimen. Equivalent fracture toughness
K_{Jq} values obtained using Equation 6, as well as tearing modulus (T) data, are also included in Table 9.

As evident from Table 9, only two tests met all the validity criteria required by ASTM E 1820 for J_{Q} to be equivalent to J_{IC}. The majority of specimens that failed the validity criteria failed the crack extension prediction requirement, which states the final crack length measured from the fractured specimen must be within 0.635 mm of the final crack length as predicted from the final unload of the specimen at the end of the test. The final crack length measurement can exceed this difference for several reasons, including unstable crack growth at the end of the test or crack closure effects due to the rough fracture surface of the castings.

An additional requirement failed by several specimens was the crack front straightness criteria. ASTM E 1820 states the final crack front must not deviate more than 0.71 mm. However, as will be described later, because these castings have relatively large grain sizes, the crack front can be quite irregular. Specifically, the crack path may propagate across large prior beta grains or alpha colonies that would cause the crack front to be irregular. An example of a specimen that failed the final crack front straightness criteria is shown in Figure 47 where a very irregular crack front (Δa > 0.71 mm) is evident for the final tensile crack growth region.

Such crack growth across large prior beta grains or alpha colonies may lead to a reduction in the fracture initiation toughness (J_{Q} values). As calculated in the previous section, if approximately 20% of the grains at the original crack front are oriented such that the crack readily propagates across them, the specimen compliance will decrease such that it will appear the entire crack front grew 0.2 mm,
at which point $J_Q$ is measured. As shown in Figure 47, the crack path is likely to follow microstructure features with low resistance to crack growth.

Although it is common for cast metals to fail the crack front straightness requirements due to preferential cracking through certain microstructure features, errors in the final crack extension prediction do not strongly affect the $J_Q$ value because $J_Q$ is measured at only 0.2 mm of crack extension. Additionally, the $J_Q$ values from specimens with non-valid data correlate well with those that did develop valid $J_Q$ values. Thus, although many of the cast fracture toughness specimens did not pass all the validity criteria to allow for a valid $J_{IC}$, the $J_Q$ values will be assumed to be reliable indicators of the dependence of toughness on heat treatment and microstructure.
Table 9: Fracture Toughness Properties for Cast Ti-5111

<table>
<thead>
<tr>
<th>Plate ID *(heat treatment)</th>
<th>Specimen</th>
<th>J_\alpha (kJ/m^2) (ASTM E 1820)</th>
<th>J_\alpha (kJ/m^2) (1 grain size)</th>
<th>K_{Jq} (MPa m^{1/2}) (ASTM E 1820)</th>
<th>T</th>
<th>Valid J_{IC} (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GMC 1 (as-cast &amp; HIPed)</td>
<td>1</td>
<td>118</td>
<td>168</td>
<td>123</td>
<td>5.2</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>127</td>
<td>168</td>
<td>128</td>
<td>2.8</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>96</td>
<td>151</td>
<td>111</td>
<td>8.6</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>150</td>
<td>175</td>
<td>139</td>
<td>2.8</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>125</td>
<td>165</td>
<td>127</td>
<td>4.2</td>
<td>N</td>
</tr>
<tr>
<td>GMC 2 (β, AC)</td>
<td>1</td>
<td>120</td>
<td>168</td>
<td>124</td>
<td>8.0</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>100</td>
<td>168</td>
<td>113</td>
<td>6.2</td>
<td>N</td>
</tr>
<tr>
<td>GMC 3 (β, FC)</td>
<td>1</td>
<td>96</td>
<td>154</td>
<td>111</td>
<td>4.9</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>N</td>
</tr>
<tr>
<td>GMC 4 (β, AC, α/β, AC)</td>
<td>1</td>
<td>89</td>
<td>165</td>
<td>107</td>
<td>15.0</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>113</td>
<td>175</td>
<td>121</td>
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<td>3</td>
<td>151</td>
<td>210</td>
<td>139</td>
<td>8.3</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>122</td>
<td>193</td>
<td>125</td>
<td>12.0</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>132</td>
<td>193</td>
<td>131</td>
<td>9.9</td>
<td>N</td>
</tr>
<tr>
<td>GMC 5 (β, FC, α/β, FC)</td>
<td>1</td>
<td>146</td>
<td>180</td>
<td>137</td>
<td>5.4</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>112</td>
<td>175</td>
<td>120</td>
<td>16.2</td>
<td>N</td>
</tr>
<tr>
<td>GMC 6 (α/β, AC)</td>
<td>1</td>
<td>138</td>
<td>228</td>
<td>133</td>
<td>7.0</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>135</td>
<td>214</td>
<td>132</td>
<td>11.8</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>143</td>
<td>210</td>
<td>136</td>
<td>6.7</td>
<td>Y</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>116</td>
<td>184</td>
<td>122</td>
<td>9.5</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>122</td>
<td>184</td>
<td>125</td>
<td>8.9</td>
<td>N</td>
</tr>
<tr>
<td>GMC 7 (α/β, FC)</td>
<td>1</td>
<td>78</td>
<td>182</td>
<td>100</td>
<td>20.3</td>
<td>N</td>
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<tr>
<td></td>
<td>2</td>
<td>78</td>
<td>172</td>
<td>100</td>
<td>9.3</td>
<td>N</td>
</tr>
<tr>
<td>IC 1 (β, FAC)</td>
<td>1</td>
<td>54</td>
<td>127</td>
<td>76</td>
<td>9.0</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>45</td>
<td>116</td>
<td>83</td>
<td>7.0</td>
<td>Y</td>
</tr>
<tr>
<td>IC 2 (β, FAC, α/β, AC)</td>
<td>1</td>
<td>100</td>
<td>200</td>
<td>113</td>
<td>15.1</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>108</td>
<td>219</td>
<td>118</td>
<td>10.8</td>
<td>Y</td>
</tr>
<tr>
<td>IC 3 *(α, β, FC)</td>
<td>1</td>
<td>77</td>
<td>119</td>
<td>99</td>
<td>5.0</td>
<td>N</td>
</tr>
</tbody>
</table>

X = inaccurate test data due to severe out-of-plane cracking
*β = 1010 °C, α/β = 954 °C, ** α/β = 843 °C
*FAC = fan air cool (54 °C/min), AC = air cool (14 °C/min), FC = furnace cool (1 °C/min)
Table 10: Quantification of Critical Microstructure Features in Cast Ti-5111

<table>
<thead>
<tr>
<th>Plate ID</th>
<th>Phase Region</th>
<th>Temp (°C)</th>
<th>Cooling Rate (°C/min)</th>
<th>*VFA</th>
<th>*PBGS (µm)</th>
<th>*ACS (µm)</th>
<th>*ALT (µm)</th>
<th>*GBAT (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GMC 1</td>
<td>cast+HIP</td>
<td>900</td>
<td>2</td>
<td>0.69</td>
<td>1200</td>
<td>180</td>
<td>3.0</td>
<td>7.4</td>
</tr>
<tr>
<td>GMC 2</td>
<td>β</td>
<td>1010</td>
<td>14</td>
<td>0.62</td>
<td>1120</td>
<td>135</td>
<td>1.9</td>
<td>4.0</td>
</tr>
<tr>
<td>GMC 3</td>
<td>β</td>
<td>1010</td>
<td>1</td>
<td>0.74</td>
<td>1130</td>
<td>195</td>
<td>4.3</td>
<td>10.3</td>
</tr>
<tr>
<td>GMC 4</td>
<td>β/α/β</td>
<td>1010</td>
<td>14</td>
<td>0.78</td>
<td>920</td>
<td>120</td>
<td>3.1</td>
<td>5.3</td>
</tr>
<tr>
<td>GMC 5</td>
<td>β/α/β</td>
<td>1010</td>
<td>1</td>
<td>0.8</td>
<td>1090</td>
<td>230</td>
<td>5.1</td>
<td>14.1</td>
</tr>
<tr>
<td>GMC 6</td>
<td>α/β</td>
<td>954</td>
<td>14</td>
<td>0.76</td>
<td>1210</td>
<td>160</td>
<td>2.8</td>
<td>7.1</td>
</tr>
<tr>
<td>GMC 7</td>
<td>α/β</td>
<td>954</td>
<td>1</td>
<td>0.73</td>
<td>1370</td>
<td>220</td>
<td>3.0</td>
<td>7.1</td>
</tr>
<tr>
<td>IC 1</td>
<td>β</td>
<td>1010</td>
<td>55</td>
<td>0.56</td>
<td>1750</td>
<td>70</td>
<td>0.5</td>
<td>2.5</td>
</tr>
<tr>
<td>IC 2</td>
<td>β/α/β</td>
<td>1010</td>
<td>55</td>
<td>0.74</td>
<td>1750</td>
<td>120</td>
<td>2.0</td>
<td>4.8</td>
</tr>
<tr>
<td>IC 3</td>
<td>α/β</td>
<td>843</td>
<td>1</td>
<td>0.84</td>
<td>1800</td>
<td>215</td>
<td>4.0</td>
<td>5.6</td>
</tr>
</tbody>
</table>

VFA = volume fraction alpha
PBGS = prior beta grain size
ACS = alpha colony size
ALT = alpha lath thickness
GBAT = grain boundary alpha thickness
Figure 47: Fracture surface of fracture toughness specimen GMC 6-5 (α/β anneal at 954 °C, air cool) showing an irregular crack front
As shown in Table 9, the fracture toughness data for the graphite mold castings range from a low of 78 kJ/m² for GMC 7 to a high of 151 kJ/m² for a cast GMC 4 specimen. Recall from Chapter 4 that the microstructure of GMC 7 (alpha/beta annealed at 954 °C and furnace cooled) is very similar to its faster cooled counterpart, casting GMC 6 (alpha/beta annealed at 954 °C and air-cooled). However, the fracture toughness developed from these seemingly similar microstructures is quite different as GMC 6 consistently achieved fracture toughness values well above 100 kJ/m² (ranging from 116 kJ/m² to 143 kJ/m²) while $J_Q = 78$ kJ/m² for GMC 7. Reasons for this difference in fracture initiation toughness are discussed in detail in Section 6.3.

Aside from GMC 6, both GMC 1 (as-cast and HIPed) and GMC 4 (beta anneal at 1010 °C, air cool, alpha/beta anneal at 954 °C, air cool) exhibited high toughness values. With the exception of one specimen each, both castings exhibited $J_Q > 100$ kJ/m². For these three “high toughness” conditions, five specimens were tested, and Table 11 shows the average and standard deviations for both fracture initiation toughness (measured using ASTM E 1820 and after crack growth equal to one grain size) and tearing modulus for the three conditions. The data is displayed graphically in Figure 48, where it is evident that GMC 4 and GMC 6 provide the best combinations of fracture initiation toughness and resistance to crack propagation with average fracture toughness values of 122 kJ/m² and 130 kJ/m², respectively, and average tearing moduli of 11 and 9, respectively. Although the fracture initiation toughness of as the as-cast GMC 1 is relatively good, its resistance to crack propagation (tearing modulus) is low.
Table 11: Statistical Analysis of the Toughness and Tearing Modulus Values from Tests of Selected Castings

<table>
<thead>
<tr>
<th>Fracture Toughness Property</th>
<th>GMC 1</th>
<th>GMC 4</th>
<th>GMC 6</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>J_Q</strong> (ASTM E 1820)</td>
<td>Average</td>
<td>123</td>
<td>122</td>
</tr>
<tr>
<td></td>
<td>St. Dev</td>
<td>19</td>
<td>23</td>
</tr>
<tr>
<td><strong>J_Q</strong> (@ 1 grain size)</td>
<td>Average</td>
<td>165</td>
<td>187</td>
</tr>
<tr>
<td></td>
<td>St. Dev</td>
<td>9</td>
<td>18</td>
</tr>
<tr>
<td><strong>T</strong></td>
<td>Average</td>
<td>5</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>St. Dev</td>
<td>2</td>
<td>3</td>
</tr>
</tbody>
</table>

Figure 48: Graph showing inverse relationship between fracture toughness (measured using both the ASTM E 1820 standard and at 1 grain size (GS)) and tearing modulus for Ti-5111 castings
Given the previously discussed sensitivity of $J_Q$ to easy crack growth within only a few grains, $J_Q$ values calculated at a crack length equal to one full grain size for each sample set are also included in Table 11. GMC 1 showed a large decrease in the standard deviation, probably because once the entire crack grows the length of a grain, grain size and alpha colony size effects become less apparent. However, GMC 4 and GMC 6, which have much higher tearing moduli, do not show the expected decrease in standard deviation for unknown reasons.

For both measurements of $J_Q$, Figure 48 shows an inverse relationship between the fracture initiation toughness ($J_Q$) and the tearing modulus ($T$). Although this relationship is not fully understood, a possible explanation involves the influence of grain and/or alpha colony orientations at the transition between the fatigue pre-crack and tensile crack growth regions. If several grains are oriented to promote easy crack growth, the fracture initiation toughness will be low. The corresponding tearing modulus will be relatively high because it will take a significant amount of additional energy to propagate the crack front through the remaining grains. On the other hand, if very few grains crack easily at the onset of tensile crack growth, large amounts of energy will be required to cause the entire crack front to move forward. Once the crack begins to grow, enough energy has been put into the system to allow the crack to continue growing relatively easily, thereby leading to a high $J_Q$ value and a low $T$ value.

Despite the similarities in crack initiation and growth resistance between GMC 4 and GMC 6, they were heat-treated differently, and thus, developed somewhat different microstructures. Both castings were air-cooled after their respective heat
treatments; however, GMC 4 underwent a duplex anneal (beta anneal at 1010 °C, air cool, alpha/beta anneal at 954 °C, air cool) while GMC 6 received only an alpha/beta anneal at 954 °C. As Table 4 showed, both castings developed alpha laths of similar thickness (ALT = 3.1 µm for GMC 4 and ALT = 2.8 µm for GMC 6). The alpha colony size, prior beta grain size, and grain boundary alpha thickness were all approximately 30% larger in the casting receiving only an alpha/beta anneal (GMC 6). Specifically, the castings had alpha colony sizes of 120 µm for GMC 4 and 160 µm for GMC 6, average prior beta grain sizes of 920 µm for GMC 4 and 1210 µm for GMC 6, and grain boundary alpha thicknesses of 5.3 µm and 7.1 µm, respectively. Thus, given an expected influence of prior beta grain size / alpha colony size, the similarities between crack initiation and growth behavior of these two conditions is somewhat surprising. It is possible that the beneficial effects of large prior beta grains and alpha colonies was somewhat negated by a thick grain boundary alpha layer that promoted grain boundary fracture.

A better understanding of the influence of alpha colony size on fracture initiation toughness and crack growth can be obtained by comparing two castings with rather different alpha colony sizes, but with similar other microstructure features. As mentioned previously, the microstructure of GMC 7 (alpha/beta anneal at 954 °C, furnace cool) is similar to GMC 6 (alpha beta anneal at 954 °C, air cool), particularly the alpha lath thickness and grain boundary alpha thickness. However, a significant difference in alpha colony size exists, with GMC 6 having an average alpha colony size of 160 µm and GMC 7 having an average alpha colony size of 220 µm. The J_0 values for GMC 6 are all above 115 kJ/m^2 while GMC 7 has J_0 values of
only 78 kJ/m$^2$. This comparative analysis between GMC 6 and GMC 7 suggests increasing alpha colony size may decrease the fracture initiation toughness ($J_\alpha$) of graphite mold cast Ti-5111. Similarly, larger prior beta grain sizes will likely decrease fracture initiation toughness because the grain size controls the maximum size of the alpha colonies. Thus, if the prior beta grain size increases, the alpha colony size may also increase.

With regards to the fracture behavior of the investment castings, the graphite mold castings performed better than the investment castings, with the exception of casting IC 2, which achieved fracture toughness values around 100 kJ/m$^2$. A likely cause for the reduced crack initiation toughness in the investment cast specimens is the role of a very large prior beta grain (average PBGS = 1750 µm) on the crack initiation toughness parameter $J_\alpha$ (this grain size averages 500 – 600 µm larger than most of the graphite mold specimens). As discussed in Section 6.1, cracking of only a few large grains will satisfy the crack advance requirement of ASTM E 1820.

An additional cause for the reduced fracture initiation toughness for investment casting IC 1 (beta anneal, fan-air cool) is the fine microstructure developed. As detailed in Table 10, IC 1 exhibited alpha laths 0.5 µm in thickness, alpha colonies 70 µm in size, and a grain boundary alpha phase only 2.5 µm thick. Each of these microstructure features was much smaller than any of the other castings; see Figure 49, which contrasts the low toughness specimen IC 1 and the high toughness specimen GMC 4. As reported in the literature [39,38], a fine alpha lath structure degrades fracture toughness in titanium alloys. Typically, a coarser
lath structure is desirable to achieve higher fracture toughness values due to increased crack deflection.

Finally, the reduced fracture initiation toughness in the furnace cooled investment casting (IC 3 − alpha/beta anneal at 843 °C, furnace cool) may also relate to the discontinuity of the beta phase. All of the graphite mold castings, as well as investment casting IC 2 (beta anneal at 1010 °C, fan-air cool, alpha/beta anneal at 954 °C, air cool) showed continuous beta laths; however, IC 3 exhibited discontinuous beta laths, see Figure 50. Chan [75] and Soboyejo [76] have shown a continuous beta phase enhances fracture initiation toughness. The benefits of the ductile phase on crack initiation toughness are only achieved when the beta phase is continuous. An in-depth discussion on mechanisms enhancing fracture initiation toughness is presented in Section 6.3.

In summary, the graphite mold castings exhibited higher crack initiation and crack propagation resistance than the investment castings. The alpha colony size and to an extent prior beta grain size appears to play the most significant role in controlling the fracture toughness of the graphite mold castings such that increasing colony size decreases the crack initiation toughness. The graphite mold castings GMC 4 (beta anneal at 1010 °C, air cool, alpha/beta anneal at 954 °C, air cool) and GMC 6 (alpha beta anneal at 954 °C, air cool) demonstrated the best combination of crack initiation toughness (JIC) and crack propagation resistance (T). Of the investment castings, IC 2 (beta anneal at 1010 °C, fan-air cool, alpha/beta anneal at 954 °C, air cool) exhibited the highest crack initiation toughness and crack propagation resistance. The fracture initiation toughness of the other investment
castings was limited by the influence of very large prior beta grains, a fine microstructure (i.e. thin alpha laths and grain boundary alpha), and discontinuous beta phase; see Figure 49 and Figure 50 for contrasting microstructures.
Figure 49: Micrographs contrasting (a) the low fracture toughness specimen IC 1 ($J_Q = 45 \text{ kJ/m}^2$) with a fine microstructure to (b) the high fracture toughness specimen GMC 4 ($J_Q = 122 \text{ kJ/m}^2$) with a relatively coarse microstructure.
Figure 50: Micrographs contrasting (a) the low toughness ($J_Q = 77 \text{ kJ/m}^2$) specimen IC 3 ($\alpha/\beta$ anneal at 843 °C, furnace cool) showing discontinuous beta phase and (b) the high toughness ($J_Q = 108 \text{ kJ/m}^2$) specimen IC 2 ($\beta$ anneal at 1010 °C, fan-air cool, $\alpha/\beta$ anneal at 954 °C, air cool) showing continuous beta phase.
6.3 Effects of Intrinsic Toughening on Crack Initiation Behavior

The influence of the microstructure on the initiation and growth of cracks in the cast Ti-5111 alloy can be described in terms of the intrinsic and extrinsic toughening mechanisms in the material. Intrinsic toughness through matrix slip and crack tip blunting influences the crack initiation ($J_{IC}$) behavior while extrinsic toughening through such processes as crack bifurcation, crack deflection, ductile phase bridging, shear ligament toughening, and microcrack shielding affect the resistance curve (tearing modulus, $T$) behavior (and to a lesser extent $J_{IC}$). This section considers the microstructure effects on the intrinsic toughening mechanisms in the cast Ti-5111 alloy, and the microstructure features that limit initiation toughness.

Crack initiation behavior of metals is strongly affected by the ductility of the matrix material. When the matrix has good ductility, the crack tip will become blunted; and thus, more energy will be required to advance the crack. In many titanium alloys, common fracture modes include slip-induced cracking along planar slip bands or decohesion of alpha grain boundaries or alpha colony intersections [77,78,79]. Based on the discussion presented in the previous chapter on the tensile behavior of cast Ti-5111, the current alloy exhibits similar fracture modes. This type of fracture behavior is consistent with a material having an insufficient number of slip systems (i.e. hcp alpha phase) to accommodate plastic incompatibility strains developed between phases or grains [80]. By having a relatively continuous network of ductile beta phase in the material, plastic deformation can occur to a greater extent prior to cracks initiating at alpha colony boundaries, slip bands, or
along the grain boundary alpha phase. As the beta phase deforms, the crack blunts and strain accumulates at the crack tip prior to crack advancement; thereby leading to a higher initiation toughness.

As discussed previously in Section 6.1, the fracture initiation toughness for GMC 6 (alpha/beta anneal at 954 °C, air cool) and GMC 7 (alpha/beta anneal at 954 °C, furnace cool) was rather different even though their microstructures were similar. It is believed that the differences in alpha colony size played a role in the differences in fracture toughness. In this section, specimens from both GMC 6 and GMC 7 are examined to understand better the role of microstructure in controlling fracture initiation toughness.

Specimens GMC 6-2 ($J_Q = 135 \text{ kJ/m}^2$) and GMC 7-2 ($J_Q = 78 \text{ kJ/m}^2$) were chosen for examination because of their large difference in $J_Q$ values. Initial examination of the fracture surfaces revealed secondary cracking at the transition between the fatigue pre-crack region and the tensile crack growth region in specimen GMC 6-2 (shown in Figure 51a) while specimen GMC 7-2 showed regions of smooth, easy crack growth between the fatigue pre-crack and the tensile crack growth regions (shown in Figure 51b). Each of these specimens was cross-sectioned, polished, removed from its mount, and examined to correlate the fracture surface with the underlying microstructure.
Figure 51: The fracture surface of (a) the high fracture toughness specimen GMC 6-2 ($J_q = 135 \text{ kJ/m}^2$) and (b) the low fracture toughness specimen GMC 7-2 ($J_q = 78 \text{ kJ/m}^2$)
The cross-section of the high-toughness specimen GMC 6-2 is shown in Figure 52. The transition between the fatigue pre-crack and tensile crack growth region was verified by (1) examining the fracture surface at high magnification in the SEM to determine the presence and location of fatigue striations and (2) macro examination of the heat-tinted fracture surface using the stereo microscope, which clearly shows the transition between the pre-crack and tensile crack growth regions. The fracture surface of GMC 6-2 shows a large secondary crack directly at the end of the fatigue pre-crack (Figure 52).

Additionally, directly below the fracture surface at the initiation of tensile crack growth, voids have formed in the high toughness specimen; see Figure 53. The formation of voids below the fracture surface, along with the out-of-plane cracking, suggest significant crack-tip blunting occurred that led to increased strain accumulation at the crack tip and an enhancement in the material’s fracture initiation toughness. The severity of blunting that occurs at the crack tip is directly proportional to the process zone size ahead of the crack tip. As shown schematically in Figure 54, the process zone is a small region inside the plastic zone, and is located directly ahead of the crack tip. This region is characterized by high strains developed at the crack tip. The size of the process zone is approximately twice the crack tip opening displacement (2CTOD) [81,82]. Therefore, for voids to form below the fracture surface and directly at the transition of tensile crack growth, the voids should have formed within the process zone of the material. The distance from the fracture surface to the voids shown in Figure 53 (~250 µm) is approximately equal to the radius of the process zone. Therefore, if the
radius of the process zone $\approx 250 \, \mu m$, the process zone is $\approx 500 \, \mu m$ in size. Thus, based on the process zone size being approximately equal to 2CTOD, the crack tip opening displacement is approximately 250 $\mu m$ in size. Such a large CTOD implies significant crack tip blunting occurred prior to initial crack extension during tensile crack growth.

**pre-crack / stable crack growth transition**

**secondary cracking**

*Figure 52: Fracture surface profile of cross-sectioned fracture toughness specimen GMC 6-2*
Figure 53: Enlargement of the fatigue pre-crack / tensile crack growth transition region showing secondary cracking and large void formation within a single alpha colony
Figure 54: Schematic representation of the stress distribution, process zone, and plane strain plastic zone associated with the crack tip (adapted from Ewalds and Wanhill [83])
The previous example showed the influence of crack tip blunting on enhancing the intrinsic toughness (and thus, crack initiation resistance) of GMC 6-2. However, the question remains as to why GMC 7 did not exhibit good crack initiation toughness. The cross-section of the low-toughness specimen GMC 7-2 is shown in Figure 55a. At this particular location on the fracture surface, the fracture profile shows a rather smooth fracture surface characterizing both the pre-crack and tensile crack growth regions. An examination of the microstructure below the fracture surface shows the fatigue pre-crack growing through a single large alpha colony (Figure 55b). At the onset of tensile crack growth along this section of the crack front, very little energy was required to enable the crack to continue growing through the same alpha colony. In this case, the crack grew approximately 500 microns before it encountered another alpha colony and changed direction. Recall that the fracture toughness specimen is 18 - 20 mm in thickness, and per ASTM E 1820, the fracture toughness is measured at 0.2 mm of crack growth. Therefore, if sections along the crack front grow approximately 0.5 – 1 mm easily, the compliance of the specimen will decrease as if the entire crack grew 0.2 mm, thereby reducing the initial fracture toughness value ($J_{IC}$).

When the crack growth path is such that the crack grows easily through a large microstructure feature, intrinsic toughening due to crack tip blunting does not occur. Instead, the crack remains relatively sharp and begins to grow at lower strain accumulations when a tensile load is applied. The cross-section of specimen GMC 7-2 did not exhibit cracking or void nucleation below the fracture surface, indicative of a small process zone and little strain accumulation prior to crack growth.
Figure 55: Fracture surface profile of the low toughness specimen GMC 7-2 showing (a) rough fracture surface and crack path and (b) transition between fatigue pre-crack and tensile crack growth occurred within single alpha colony
6.4 Effects of Extrinsic Toughening Mechanisms on Tearing Modulus and Crack Propagation

Extrinsic toughening mechanisms that can enhance a material's resistance to crack propagation include the following: crack deflection, crack bifurcation, shear ligament toughening, and ductile-phase bridging. Each of these extrinsic toughening mechanisms plays a role in enhancing the crack propagation resistance (tearing modulus) of cast Ti-5111. Crack deflection, crack bifurcation, and shear ligament toughening are all related to the development of a tortuous crack path in the material [75,78,80]. This section first examines the relationship between extrinsic toughening mechanisms, surface roughness, and tearing modulus of the cast Ti-5111 alloy.

The ease of crack propagation for each casting is directly related to the tearing modulus. A lower tearing modulus implies continued crack growth occurs without significant increases in stress or energy. Previous studies of crack growth behavior in titanium alloys have associated crack growth resistance with crack path tortuosity [25,75,80]. A rough fracture surface is evidence of a tortuous crack path, and as crack tortuosity increases, the resistance to crack propagation, and thus the tearing modulus, also increases. In the present study, the surface roughness of six fracture toughness specimens (GMC 1-1, GMC 1-2, GMC 6-2, GMC 7-1, GMC 7-2, and IC 1-2) was measured to correlate fracture surface roughness with the tearing modulus. As shown in the surface roughness map for specimen GMC 6-2 (Figure 56), there are distinct changes in roughness between the machined notch, fatigue pre-crack, stable crack growth, and final fracture regions for an area approximately 15 mm by 9 mm. As expected, the machined notch has the smoothest surface, the
fatigue pre-crack region is slightly rougher, the stable crack growth region for this particular specimen is rather rough, and the final fracture region is the roughest.

The surface roughness for each of the other specimens was measured across a region that was directly ahead of the fatigue pre-crack and that is approximately 20 mm by 3 mm wide. This area encompasses the region of tensile crack growth where the tearing modulus is measured (from 0.2 mm to 2.5 mm of crack growth). Surface roughness maps for each specimen were developed, and those for specimens GMC 1-1 and GMC 7-2 are shown in Figure 57. Specimen GMC 1-1 had a relatively low tearing modulus of 5.2 while specimen GMC 7-1 had a relatively high tearing modulus of 20.3. As the maps indicate, the roughness of these two specimens is significantly different, with specimen GMC 7-1 exhibiting a much rougher surface.

The difference in surface roughness and corresponding difference in tearing modulus between specimens GMC 1-1 and GMC 7-1 suggest the extrinsic toughening mechanisms that influence surface roughness (crack deflection, crack bifurcation, and shear ligament toughening) may be enhancing the crack growth resistance of GMC 7-1. One of the easiest ways for a crack to absorb energy and resist growth is for a crack to deviate in different directions. For example, the roughness map for specimen GMC 7-1 in Figure 57 shows that near the left of the specimen the crack was deflected down to a z-height of approximately 200 µm and up to a z-height of approximately 3000 µm. The process of a crack being deflected or bifurcating in two opposite directions reduces the local stress intensity factor by shielding the crack tip [75]. Chan [75,78] has shown that the toughening effect from
crack deflection is most pronounced as the angle of deflection increases; for example, for Ti-24Al-11Nb, a crack deflection angle of 60 degrees will cause a 25 percent increase in toughness.

In addition to the toughness enhancement by crack deflection in GMC 7-1, shear ligament toughening also improved the crack growth resistance. When the crack mentioned previously bifurcated, it formed a height difference of approximately 2800 µm between the two new cracks. For the main crack to move forward, it had to join these two ligaments. Tearing 2800 µm of material to join the ligaments required a great deal of energy, thereby increasing the tearing resistance of the material through shear ligament toughening.
Figure 56: Surface roughness profile of machined notch, fatigue pre-crack, tensile crack growth, and final fracture of specimen GMC 6-2
Figure 57: Surface roughness maps for graphite mold cast specimen GMC 1-1 (T = 5.2) and GMC 7-1 (T = 20.3)
A comparison of the surface roughness distributions for the series of six fracture toughness specimens are plotted in Figure 58. At each point measured (approximately 150,000 data points), the specific z-height was determined, which is presented graphically in Figure 58. As the graph shows, a significant difference is evident in the roughness curves developed from specimens GMC 1-1 and GMC 7-1, which had tearing modulus values of 5.2 and 20.3, respectively.

To achieve a more quantitative estimate of the roughness of the samples, the roughness distribution curves shown in Figure 58 were fit using peak fitting software to extract the main characteristics of the sample roughness: average roughness, standard deviation, and whether the distribution was bimodal. The curves shown were fit using PeakFit software.\(^1\) PeakFit allows a manual or automatic fit of the roughness profiles to minimize the error. The fittings were performed using a Pearson VII peak shape (a peak shape that combines both Lorentzian and Gaussian characteristics). The curve peak (corresponding to the z-height of greatest frequency) \((z_i)\), intensities (area), full width half maximum \((\sigma)\) and tails can all be independently varied to achieve the best fit. The decision for the number of peaks to use is also done manually (visually) by judging whether the peak shape fits.

An example of such a fit computed for specimen GMC 7-2 is shown in Figure 59. Using two peaks to fit this particular data set, very good agreement is reached, and the peak characteristics are shown in Figure 59. To find the average surface roughness, we perform the calculation that weights the surface roughness contributions:

\[ \text{Average Roughness} = \sum \left( \text{Contribution} \times \text{Height} \right) / \sum \text{Contribution} \]

\(^1\) PeakFit version 4.06, Systat Software, 1995.
\[ \bar{z} = (z_1 \times area_1) + (z_2 \times area_2) \]

\[ \bar{z} = (1.21 \times 0.88) + (0.97 \times 0.12) = 1.18 \]

This was done for all the samples studied, and the average surface roughness values, along with the correlating tearing modulus, are shown in Figure 60.

As the graph in Figure 60 shows, tearing modulus increased as surface roughness increased. This result is expected because a rougher fracture surface develops as a result of crack path tortuosity. A tortuous crack path is indicative of extrinsic toughening, where crack deflection, crack bifurcation, and shear ligament toughening reduce the local stress field at the crack tip and absorb more energy by increasing the crack length. Thus, crack growth resistance, tearing modulus, and surface roughness are all enhanced.
Figure 58: Quantitative comparison of the roughness curves for each fracture toughness specimen measured.

Figure 59: Example of peak fit for specimen GMC 7-2. Two Pearson VII peaks are used to fit the fracture surface roughness distribution.
Figure 60: Relationship between fracture surface roughness and tearing modulus (crack propagation)
The next issue is to correlate the extrinsic toughening mechanisms and the surface roughness behavior in Figure 58 with the microstructure of cast Ti-5111. Shear ligament toughening is a common extrinsic toughening mechanism in lamellar titanium alloys [78,79,80,84]. Chan [78,80,84] observed the formation of shear ligaments due to mismatched crack planes in Ti-24Al-11-Nb and Ti-47Al-2.6Nb-2(Cr+V), and Deve et al. [79] observed similar behavior in Ti-48Al-2Nb-2Cr. In the studies by Chan, crack growth during monotonic tensile loading of a fracture toughness specimen was videotaped and photographed. Five shear ligaments were observed ahead of the crack tip due to microcracking in different alpha colonies. It was shown in Chapter 5 of this study, along with other titanium studies [35,70,85], that deformation and crack propagation often occurs on preferential slip planes. Therefore, it is likely that cracking will occur on planes of preferred orientation in alpha colonies ahead of the main crack. A schematic illustration of potential shear ligament formation in cast Ti-5111 is shown in Figure 61.
Figure 61: Schematic illustration of shear ligaments joining cracks that formed ahead of main crack.
The high degree of roughness on the fracture surfaces of cast Ti-5111 (up to 3 mm change in z-height based on Figure 58) suggests the crack path may be controlled by the large microstructure features, particularly prior beta grain size and alpha colony size. These features may be deflecting cracks or fracturing in a manner to promote shear ligament toughening. To correlate the microstructure with the fracture path, GMC 7-2 ($J_\alpha = 78 \text{ kJ/m}^2$, $T = 9.3$) and GMC 6-2 ($J_\alpha = 135 \text{ kJ/m}^2$, $T = 11.8$) were examined in detail. The fracture surface profile and underlying microstructure of specimen GMC 7-2 is shown in Figure 62. The figure shows that the crack path during tensile crack growth is tortuous, propagating within alpha colonies (Figure 62b & Figure 63) and occasionally adjacent grain boundary alpha, shown in Figure 62c. Observations of the crack path for both GMC 7-2 and GMC 6-2 showed fracture often occurred parallel to alpha laths, as shown in Figure 63. It is likely crack growth was relatively easy parallel to the alpha laths and along the grain boundary alpha in Figure 62b and Figure 62c, while the alpha colony separating them was fractured by shear. If this occurred, the energy required to tear the ligament between these two potential cracks would be rather high and increase the material’s tearing resistance.
fracture parallel to alpha laths

possible shear ligament fracture

grain boundary alpha (see Figure 62c)

transition to tensile crack growth
Figure 62: Cross-sectioned view of fracture toughness specimen GMC 7-2 showing (a) rough fracture contour (b) low-energy fracture path parallel to alpha laths and along grain boundary alpha, and potential shear ligament fracture (c) fracture along grain boundary alpha phase. (In the polarized light micrographs in (a) and (b), each color/shade of gray represents a different alpha colony due to differences in alpha lath orientation)
The dominating microstructure feature controlling crack deflection appears to be alpha colonies. As the crack grew through the microstructure of both GMC 6-2 and GMC 7-2, it often chose to follow alpha colonies propagating both parallel and perpendicular to the laths. Once it reached a new alpha colony, the crack was often deflected such that it followed parallel to the alpha laths of the new colony. Figure 63 shows that as the crack grew parallel to the alpha laths, it was deflected in a new colony to grow parallel to laths of different orientation. Similarly, specimen GMC 6-2 showed the main crack traveled approximately 500 μm out-of-plane when it intersected a new alpha colony (Figure 64).
The previous examples have shown that a growing crack will deviate around alpha colonies, with crack growth parallel to alpha laths. Therefore, increasing the alpha colony size should improve crack propagation resistance because larger alpha colonies will increase crack deflection, and thus, the surface roughness and tearing modulus.
Figure 64: Crack path being deflected by an alpha colony, which was approximately 500 µm in size.
The final form of extrinsic toughening evident in cast Ti-5111 is ductile phase bridging. As suggested by its name, ductile-phase bridging can promote crack growth resistance by bridging the gap between microcracks that have formed in a less-ductile phase. For example, in titanium lath structures, the beta phase between the alpha laths is considered more ductile than the alpha laths because it has more slip systems to accommodate plastic deformation. Therefore, if cracks were to form on slip planes through the alpha phase, the more ductile beta phase will resist tearing until additional stresses are applied. The ductile beta phase would reduce the crack opening displacement and lower the near-tip stress intensity of the main crack. An example of ductile phase bridging in a cast Ti-5111 fracture toughness specimen is provided in Figure 65. The figure shows regions of cracking on both sides of the beta phase, but the beta phase remains intact. A typical resulting fracture surface of such an area is shown in Figure 66. The image shows low energy fracture associated with the alpha laths, and ductile tearing of the beta phase. Therefore, it is likely the alpha laths fractured prior to the more ductile beta phase. Although this type of toughening mechanism is not expected to improve the crack growth resistance and tearing modulus of cast Ti-5111 as significantly as the crack deflection, crack bifurcation, and shear ligament toughening processes, ductile phase bridging is still a useful mechanism to enhance crack growth resistance.
Figure 65: Ductile phase bridging of the beta phase in specimen GMC 7-2.

Figure 66: Stereo-section image of specimen GMC 7-2 showing low-energy fracture of individual alpha laths and ductile tearing of the beta phase.
Finally, it should be noted that the intrinsic toughening mechanism of crack-tip blunting, which was described previously in the discussion on crack initiation, also influences the crack propagation behavior. It was suggested in Section 6.3 that the presence of voids below the fracture surface is indicative of a large process zone and crack-tip blunting. Such voiding is observed in specimen GMC 6-2 (see Figure 67 and Figure 68) after 500 µm to 1 mm of crack growth. Several large voids (on the order of 100 µm in length) and numerous smaller ones are observed along the grain boundary alpha phase in Figure 67, and the example in Figure 68 shows voids forming on the beta phase next to the crack plane. The formation of these voids suggests the presence of a large process zone and significant crack-tip blunting. It is likely that a continuous process of crack blunting, microcracking, and crack growth occurred as the crack propagated.
Figure 67: Cross-section view from GMC 6-2 showing voids nucleating and growing along the grain boundary alpha phase
In summary, extrinsic toughening mechanisms play a vital role in enhancing the tearing modulus of cast Ti-5111. Crack deflection, crack bifurcation, and shear ligament toughening are the mechanisms that play the largest role in enhancing the crack growth resistance of the alloy. Each of these mechanisms leads to an increase in the fracture surface roughness of the material, which correlates to a larger tearing modulus.

Alpha colonies appear to be the dominating microstructure feature in controlling crack propagation. Examples were provided showing crack deflection within alpha colonies that led to tortuous crack paths and a high surface roughness. Therefore, larger alpha colonies lead to greater crack deflection, which significantly increases the material’s resistance to crack growth.
6.5 Comparison of Fracture Toughness Behavior of Cast and Hot-Rolled Ti-5111

Understanding the differences in the fracture toughness behavior between cast Ti-5111 and wrought Ti-5111 leads to a better understanding of how the microstructure features influence crack initiation and growth. Based on this understanding, the cast product can be optimized to enhance its fracture initiation toughness ($J_{IC}$) and resistance to crack growth ($T$).

The fracture toughness of wrought Ti-5111 was measured in a previous study conducted by Roe [86], and the results obtained from that study are presented in Table 12. As discussed previously, the wrought product was rolled in the beta regime and subsequently annealed in the alpha/beta regime at 954 °C followed by air cooling. As a basis for comparison to a cast microstructure, Table 12 also includes toughness data for GMC 6, which was also annealed in the alpha/beta regime at 954 °C followed by air cooling.

Table 12 shows the fracture initiation toughness ($J_Q$) and tearing modulus of the wrought plate is a function of the crack growth direction. As was discussed in Section 5.3 on the tensile properties of wrought Ti-5111, titanium alloys with a hexagonal close-packed crystal structure that have been directionally processed (i.e. hot-rolled), exhibit higher strength and ductility in the transverse direction than in the longitudinal direction. Similarly, the fracture toughness and crack growth resistance of wrought Ti-5111 are higher when loaded in the transverse direction. The specimens labeled TL signify specimen loading was transverse to the rolling direction and the crack growth was longitudinal with respect to the rolling direction.
The specimens labeled LT signify loading in the longitudinal direction and crack growth in the transverse direction with respect to the rolling direction.

As shown in Table 12, after similar heat treatments, the graphite mold casting has a significantly higher toughness than the hot-rolled plate. The average J-integral fracture toughness for the hot-rolled and annealed plate is 109 kJ/m² and 96 kJ/m² for the transverse and longitudinal specimens, respectively, while the J_Q value for the graphite mold cast material (after receiving the same heat treatment as the rolled plate) was 130 kJ/m². Table 12 also shows that the tearing moduli between the two material conditions is comparable; T = 8.2 and 6.5 for rolled plate in the transverse and longitudinal directions, respectively, while that for the casting is 9.0. Thus, the cast Ti-5111 product form exhibits better fracture initiation toughness than its wrought counterpart and slightly better crack propagation resistance.
Table 12: Comparison of Fracture Toughness Data for Wrought Ti-5111 Plate and Graphite Mold Cast Ti-5111

<table>
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<tr>
<th>Plate ID</th>
<th>Specimen</th>
<th>$J_Q$ (kJ/m$^2$) (ASTM E 1820)</th>
<th>$K_{Jq}$ (MPa m$^{1/2}$) (ASTM E 1820)</th>
<th>$T$</th>
<th>Valid $J_{IC}$ (Y/N)</th>
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</tr>
<tr>
<td>LT</td>
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<td>109</td>
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Note: Both the wrought Ti-5111 and GMC 6 cast Ti-5111 were subject to an $\alpha/\beta$ anneal at 954 °C followed by air cooling
The comparison of the fracture toughness data for the hot-rolled product form and the cast product form of Ti-5111 showed that although the materials were heat treated similarly, the casting exhibited significantly higher initiation toughness and slightly higher crack growth resistance. In examining the tensile crack growth behavior of cast Ti-5111, secondary cracking at the transition between fatigue pre-cracking and tensile crack growth often occurred. Such crack deflection can significantly increase the fracture initiation toughness in the material due to the increased energy absorbed by forming secondary cracks. For the case of the hot-rolled plate, an examination of the fracture surface shows an absence of secondary cracking at the onset of stable crack growth; see Figure 69.

Although the fracture initiation toughness of the wrought product is below that of the cast product, it remains relatively high due to significant crack-tip blunting due to the inherent ductility of the beta phase. As mentioned previously, crack tip blunting enhances crack initiation toughness by reducing local stresses at the crack tip. One method to promote crack tip blunting is the presence of a ductile matrix phase. In Ti-5111, the beta phase acts as the ductile phase that plastically deforms more easily than the alpha phase. The microstructure of the hot-rolled plate and casting GMC 6 are provided in Figure 70 for comparison. As is evident from the micrographs, the beta phase forms a continuous matrix in the hot-rolled material, but is separated by alpha laths in the cast material. Thus, the continuous ductile beta matrix in the hot-rolled plate promotes crack tip blunting by allowing the material ahead of the crack tip to deform and accumulate higher strains prior to crack initiation. Additionally, a higher volume fraction of beta phase is present in the hot-
rolled plate ($V_f \approx 65\%$) than in the casting ($V_f \approx 76\%$). Further evidence of the crack-tip blunting expected in the wrought product is the greater tensile ductility exhibited by the hot-rolled plate. As Table 8 showed, the wrought product consistently achieved elongations of 12 – 13 percent, while GMC 6 averaged 9.7 percent elongation. Therefore, the fracture initiation toughness ($J_{IC}$) of the wrought product is relatively good for high strength titanium alloys due to a continuous, ductile beta matrix and the absence of large colonies to propagate sections of the main crack easily.

Figure 69: SEM micrograph of fracture surface of fracture toughness specimen SLT-1 showing relatively smooth fracture surface with low energy fracture of elongated alpha plates
Figure 70: Microstructure of (a) wrought Ti-5111 plate showing a continuous beta matrix, which promotes crack tip blunting, and (b) microstructure of cast GMC 6 showing beta phase separated by alpha laths.
The crack propagation behavior of metals is controlled by both intrinsic and extrinsic factors. In the earlier section regarding the tearing modulus of the cast Ti-5111 alloy, the surface roughness correlation with the tearing modulus (crack propagation behavior) was influenced mostly by extrinsic toughening mechanisms such as crack deflection, crack bifurcation, and shear ligament toughening. As the crack followed large grains and alpha colonies, more energy was absorbed as the crack changed directions to follow each new colony. These extrinsic toughening mechanisms promoted a rough fracture surface where it was observed that as surface roughness increased, the material's resistance to crack growth (tearing modulus) increased.

With regards to the hot-rolled and annealed Ti-5111 plate, the crack propagation resistance was controlled almost entirely by intrinsic mechanisms. As Figure 71 and Figure 72 show, the fracture surface roughness of the wrought plate within the tensile crack growth region is much smaller than that of the cast plate receiving the same heat treatment. The fracture surface of the hot-rolled plate is comparatively smooth with no large crack path deviations off the main crack path plane. In the discussion on the crack propagation resistance of the castings, the extrinsic toughening mechanisms of crack deflection, crack bifurcation, and shear ligament toughening were shown to significantly enhance the surface roughness and crack propagation resistance (tearing modulus). It is apparent from the relatively smooth fracture surface of the wrought specimens that these extrinsic toughening mechanisms are not strong influences on the crack propagation resistance of the wrought product.
Although the surface roughness of the hot-rolled and annealed plate is small compared to the castings, the tearing modulus ranged between 5.8 and 8.7, which is fairly good crack propagation resistance. The reason the tearing modulus is still relatively good is due to the intrinsic toughening effects of a relatively ductile microstructure. The crack propagation process is a continual process of crack tip blunting and crack growth. Because wrought Ti-5111 has good ductility and significant blunting occurs at the crack tip, the tearing modulus of the material remains positive with some resistance to crack propagation.
Figure 71: Fracture surface roughness map of hot-rolled and annealed ($\alpha/\beta$ anneal at 954 °C, air cool) Ti-5111 plate specimen SLT-1

Figure 72: Fracture surface roughness map of cast Ti-5111 plate ($\alpha/\beta$ anneal at 954 °C, air cool)
Chapter 7: Conclusions & Future Work

This study examined the influence of heat treatment on the microstructure development of graphite mold cast and investment cast Ti-5111 plates as well as hot-rolled and annealed Ti-5111 plate. The effect of the resulting microstructures on the tensile behavior and the fracture toughness behavior was determined.

The following conclusions can be made regarding the microstructures developed:

- The castings all developed a lamellar alpha-beta microstructure, which was sensitive to cooling rate from the beta and alpha/beta phase regions. With an occasional exception, slowly cooled castings tended to develop larger alpha colonies, thicker alpha laths, thicker grain boundary alpha, and a higher volume fraction of alpha phase. Additionally, duplex annealed specimens (beta anneal followed by an alpha/beta anneal) also developed a coarser structure than specimens given only a beta or alpha/beta anneal due to the increased times at elevated temperatures allowing further diffusion to occur.

- The investment castings developed significantly larger prior beta grains (~1750 µm) compared to those of the graphite mold castings (~1200 µm). The larger grain size is attributed to the investment castings remaining in the beta phase region for a longer period of time after the melt was poured. In addition to the lamellar alpha-beta microstructure, the investment cast specimens that were rapidly cooled from the beta phase field at a rate of 55 °C/min developed regions of basketweave alpha.
The hot-rolled and annealed Ti-5111 plate developed coarse alpha plates in a fine transformed beta matrix that consisted of fine, acicular alpha laths within a continuous retained beta matrix.

With respect to the tensile behavior of the castings, the following conclusions are made:

- The tensile ductility of the castings was often limited by the strain required to initiate a crack. Once a large crack initiated (on the scale of the prior beta grain size or alpha colony size), the strain required to propagate the crack was relatively low leading to low ductility.

- Crack initiation typically occurred along planar slip bands that can extend through several alpha colonies and occasionally across entire prior beta grains. Thus, limiting the size of these features should inhibit the initiation of long cracks and enhance ductility.

- A secondary fracture feature noted in the tensile specimens was void formation and cracking at alpha colony boundaries; however, only minimal fracture along the grain boundary alpha phase was observed.

- The hot-rolled and annealed Ti-5111 plate showed slightly higher average tensile ductility and less scatter in the ductility values than its cast counterpart. This behavior was attributed to the wrought plate fracturing due to void nucleation, growth, and coalescence, as opposed to crack initiation and propagation in the castings.

- The yield and tensile strength of the castings was sensitive to the specimen size. Smaller diameter tensile specimens exhibited lower strengths due to the
number of large grains adjacent to the specimen surface that can subsequently yield at lower stress levels via single slip. Because tensile fracture was controlled by a crack initiation and propagation process, a significant amount of scatter occurred in the ductility values, particularly in the smallest diameter (3.2 mm) specimens. However, the average tensile ductility was not significantly affected by specimen size.

- Each of the castings exceeded the minimum ultimate tensile strength and yield strength requirements for this alloy, while the average tensile ductility was marginal.

Finally, the following conclusions were found regarding the fracture toughness and tearing modulus of cast Ti-5111:

- Determining the fracture initiation toughness ($J_{IC}$) of castings with large grain sizes poses certain issues when implementing the analysis procedures outlined in the fracture toughness test standard for metals (ASTM E 1820). Specifically, due to the large grain size of the Ti-5111 castings (average range is 920 µm – 1800 µm), only 3-5 grains would need to crack completely to give the appearance of 0.2 mm of crack extension, which is specified to determine $J_{IC}$.

- The fracture toughnesses of the graphite mold castings had a wide range of values from 78 kJ/m² (the alpha/beta annealed and slowed cooled condition) to a high of 151 kJ/m² (the duplex annealed specimen followed by air cooling). Large alpha colonies or prior beta grains oriented in such a manner to allow for easy crack initiation and growth along a significant fraction of the
crack front resulted in reduced $J_{IC}$ values. Thus, smaller alpha colonies and prior beta grains should lead to an increase in crack initiation toughness, as was the case for the casting subjected to a beta anneal plus alpha/beta anneal followed by a fast air cool.

- As a measure of the resistance to stable crack growth, the tearing modulus, $T$, was sensitive to the extrinsic toughening mechanisms of crack deflection, crack bifurcation, and shear ligament toughening; all of which promoted a rough fracture surface. As a result, the tearing modulus increased as the surface roughness of the fractured specimens increased. The alpha colonies were the dominant microstructure feature controlling crack deflection that resulted in tortuous crack paths. Therefore, an increase in alpha colony size increased the tearing modulus and crack propagation resistance of cast Ti-5111.

- When compared to hot-rolled plate after similar heat treatments, the graphite mold casting had a significantly higher initiation toughness, $J_Q$, and slightly better crack propagation resistance (tearing modulus), $T$ ($J_Q = 130 \text{ kJ/m}^2$ and $T = 9.0$ for the casting, and $J = 109 \text{ kJ/m}^2$ and $96 \text{ kJ/m}^2$ and $T = 8.2$ and $6.5$ for the hot-rolled plate loaded in the transverse and longitudinal directions, respectively). Crack initiation and growth in the hot-rolled plate was controlled mostly by crack tip blunting of the continuous beta matrix, thereby leading to relatively good resistance to crack growth despite minimal fracture surface roughness.
Future Research

Based on the results of this study, two additional projects may be considered to more fully understand the relationship between microstructure and fracture behavior in Ti-5111 as well as other near-alpha lamellar titanium alloys. First, it may be useful to determine the influence of the misorientation of adjacent alpha colonies on slip transfer between colonies. It was observed in this study that slip due to tensile loading often propagated easily through a single alpha colony and occasionally through an entire beta grain. Understanding the role of misorientation between alpha colonies on the transfer of slip would help develop a better understanding of both the crack initiation and the crack propagation behavior in cast Ti-5111. Additionally, such information could be applied to wrought Ti-5111 that has been heat treated to obtain a lamellar microstructure. Deformation-induced texture could be used to optimize specific properties of the alloy if a better understanding of orientation relationships between alpha colonies is developed.

Second, it would be useful to quantify the individual effects of the intrinsic and extrinsic toughening mechanisms in cast Ti-5111. Chan [75,77,78,80] and Soboyejo [76] developed equations to approximate the influence of toughening mechanisms such as crack deflection, crack bifurcation, shear ligament toughening, ductile phase bridging, microcrack shielding, and crack tip blunting for lamellar titanium structures. To complete such a study, imaging of the crack tip during stable crack growth would be necessary. Two possibilities include using a high resolution telescope interfaced with a video monitoring system or conducting the fracture toughness test inside a scanning electron microscope equipped with a hydraulic loading stage. Although
limited to mainly surface observations, this process would allow for measuring crack
deflection angles, shear ligament lengths between microcracks, and the length and
location of microcrack formation with respect to the crack tip and the microstructure.
By obtaining these measurements, the influence of crack deflection, shear ligament
toughening, microcrack shielding and ductile phase bridging could be quantified
using analyses developed by Rose [87], Chan [75,77,78,80], and Soboyejo [76].
Determining the magnitude of each individual toughening mechanism observed in
the crack initiation and propagation behavior of cast Ti-5111, along with other near-
alpha titanium alloys, could provide guidelines for the processing and microstructure
development of the material for critical fracture applications.
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81 Osman, T.M. and Rigney, J.D., “Introduction to the Mechanical Behavior of
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Appendix A: X-Ray Diffraction Verification of the Alpha and Beta Phases
Figure A-1: (a) X-ray diffraction plot showing alpha and beta phase formation in both GMC and IC castings, (b) JCPDS diffraction file for $\beta$-titanium, and (c) JCPDS diffraction file for $\alpha$-titanium [88]
Appendix B: Cast Ti-5111 Tensile Data

Table B-1: Additional Tensile Data for Select Casting Conditions
(GMC 1, GMC 4, and GMC 6)

<table>
<thead>
<tr>
<th>Casting</th>
<th>Specimen No.</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>0.2% Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction of Area (%)</th>
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<tr>
<td>GMC 1 (as-cast &amp; HIPed)</td>
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*AC = air cool (14 °C/min), β anneal @ 1010 °C, α/β anneal @ 954 °C
Table B-2: Tensile Properties as a Function of Specimen Diameter for Casting GMC 1 (As-Cast and HIPed)

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<th>Specimen Size</th>
<th>Specimen No.</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>0.2% Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Reduction of Area (%)</th>
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</table>
Vita
Amy C. Robinson

Education

  Thesis Title: "Optimizing Strength and Fracture Toughness of a Cast Titanium Alloy through Heat Treatment and Microstructure Control"
  Thesis Advisor: Prof. Donald Koss
  Thesis Title: "The Role of Microstructure in Controlling Fracture of AL-6XN Stainless Steel"
  Thesis Advisor: Prof. Donald Koss

Professional Experience

*Naval Surface Warfare Center, Carderock Division*, West Bethesda, MD
March 2003 – February 2007
**Materials Engineer**

- Lead engineer and program manager for 3 titanium research and analysis programs. Studied the microstructure development in cast titanium alloy Ti-5111 and cast CP2-LO and its effects on mechanical properties. Determined the mechanism of embrittlement from stress relieving Ti-5111 at specific temperature ranges previously common in industry.
- Obtained $500K in research funding from the Office of Naval Research (ONR), Missile Defense Agency (MDA), and the In-house Laboratory Independent Research (ILIR) programs to support self and 3 other engineers and technicians.
- Program manager for rhenium characterization program. Characterized pure rhenium metal by thorough microstructure examination and mechanical testing (tensile, fracture toughness, and low cycle fatigue) up to 3500°F. Quantified effects of texture developed from cold rolling vs. HIPing process that changed program’s processing of rhenium missile components.

Publications (selected)