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On the influence of microstructural features of Linear Friction Welding and Electron Beam Additive manufacturing Ti-6Al-4V on tensile and fatigue mechanical properties

by

Michael Yesid Mendoza Londono

A dissertation submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Major: Material Science and Engineering

Program of Study Committee:
Peter Collins, Major Professor
Ralph Napolitano
Richard LeSar
Mathew Frank
Jun Cui

The student author, whose presentation of the scholarship herein was approved by the program of study committee, is solely responsible for the content of this dissertation. The Graduate College will ensure this dissertation is globally accessible and will not permit alterations after a degree is conferred

Iowa State University
Ames, Iowa
2019

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DEDICATION

This research and dissertation is dedicated to my parents that with their unconditional love motivate me to set higher targets.
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<td>Linear Friction Welding</td>
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<tr>
<td>EBAM®</td>
<td>Electron Beam Additive Manufacturing</td>
</tr>
<tr>
<td>WZ</td>
<td>Welded Zone</td>
</tr>
<tr>
<td>TMAZ</td>
<td>Thermo-mechanical Affected Zone</td>
</tr>
<tr>
<td>PM</td>
<td>Parent Material</td>
</tr>
<tr>
<td>EDM</td>
<td>Electron Discharge Machining</td>
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<tr>
<td>EBSD</td>
<td>Electron Backscatter Diffraction</td>
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<tr>
<td>PED</td>
<td>Precession Electron Diffraction</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscopy</td>
</tr>
<tr>
<td>HCF</td>
<td>High Cycle Fatigue</td>
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ABSTRACT

Linear friction welding (LFW) is a solid state joining process that offers the advantage of producing aircraft components with less initial raw material. Usually, aircraft components are machined from oversized ingots to obtain the final product. This process allows the use of not-oversized ingots for welding them together to form the same final component. In addition, the inherent solid state nature of the process, eliminates all problems associated to solidification.

Various efforts have been made to evaluate such microstructure development and tensile properties associated on Ti-6Al-4V, but they have been focused on the general microstructure and not on the individual developed zones. In this study, tensile test evaluation on individual LFW zones determined yield strength of the Welded Zone (WZ) and the Thermo-mechanical Affected Zone (TMAZ) of 20 and 13% respectively greater than the Parent Material (PM) zone. Qualitative description of unindexed fraction on electron backscatter diffraction (EBSD) maps and kernel average misorientations maps (KAM) suggested a strain hardening mechanism acting. Fatigue properties are also important for aircraft components, but due to the small size of the LFW zones a conventional fatigue test was not possible. Therefore, a new ultrasonic fatigue bending test was designed using the finite element commercial package COMSOL Multiphysics®. Specific predictions on resonant frequency, shape and dimensions of specimens to evaluate individual features of a larger size than LFW such as electron beam additive manufacturing (EBAM®-Ti-6Al-4V) were given. Finally, conventional fatigue tests on EBAM®-Ti-6Al-4V specimens, showed the better performance of microstructure A compared with B under
the perspectives of crack initiation and propagation. The final idea is to use the ultrasonic approach to evaluate the small LFW zones.
CHAPTER 1. INTRODUCTION AND PROBLEM STATEMENT

1.1 Motivation

High strength, low density and good corrosion resistance are properties that make titanium alloys (e.g. Ti-6Al-4V) widely used in the aerospace industry. The high cost of titanium has limited its use in other applications like automotive industry. However, in recent years the effort to minimize the cost problem has focused into more cost-effective production methods. Linear Friction Welding (LFW) is a solid-state joining process that produces heat via friction which causes the interface material to plasticize. The main advantage of LFW resides in the fact that for aircraft structural components, oversized ingots are machined to obtain the final component, so a large amount of material is wasted. LFW allows the use of not oversized ingots for welding them together to form the same aircraft component with less waste of the initial material. However, the information regarding microstructure development of LFW is still limited, so characterization of microstructure and mechanical properties relationship has to be evaluated.

1.2 Contribution of Dissertation

This dissertation is dedicated to the following goals:

1. Evaluation of tensile test properties and their relationship with individual features of microstructure development as a consequence of the linear friction welding process on Ti-6Al-4V material.

2. Design and preliminary evaluation of ultrasonic bending fatigue test on cantilever samples to assess individual features of the microstructure of EBAM®-Ti-6Al-4V material.

3. Conventional fatigue evaluation of EBAM®-Ti-6Al-4V to use as a reference for a future validation of a complete ultrasonic fatigue test on the same material.
1.3 Dissertation Overview

This dissertation covers eight chapters. Chapter 1 provides the motivation and associated goals. In chapter 2, a brief literature review is described about linear friction welding, titanium alloys, fracture mechanics, finite element analysis, ultrasonic and conventional fatigue. Chapter 3 includes the experimental and computational techniques used in this research work. Linear friction welding zones characterization with different techniques such as SEM, EDS, EBSD and TEM-PED and tensile tests are made in Chapter 4. In chapter 5, several COMSOL Multiphysics® simulations (Finite Element Analysis or FEA) and preliminary ultrasonic fatigue tests are given. Chapter 6 provides the conventional fatigue results on EBAM®-Ti-6Al-4V specimens with analysis and discussions. Chapter 7 presents the general conclusions and suggestions for future research and Chapter 8 gives the references used in this work.
CHAPTER 2. BACKGROUND AND LITERATURE REVIEW

2.1 Physical Metallurgy of Titanium

2.1.1 Introduction

Titanium is considered to be one of the strongest metals. It has a combination of properties as high specific strength (i.e., high strength-to-weight ratio), excellent corrosion resistance and good fatigue resistance. Such characteristics enable extensive applications. However, high cost on extraction and processing limit its application [1].

The global titanium market (Fig. 2.1) demand in 2018 (375 million to 380 million pounds) is about 6% greater compared to the previous year. Higher demand in the aerospace industry and more favorable industrial and energy activity (Oil & Gas industry) will increase the demand of titanium in 8 to 10 percent in 2019 [2]. The aerospace market is an increasing pull on demand for structure and engine on commercial and military aircraft. Therefore, there is an increasing necessity to develop technology to improve the properties and reduce the production cost [3].

![Titanium industry model of demand in million pounds until 2022](image)

**Fig. 2.1** Titanium industry model of demand in million pounds until 2022 [2]
2.1.2 Phases of Titanium

Pure titanium at room temperature has a hexagonal close-packed crystal structure (α phase) with a space group symmetry P63/mmc (194). However, it has a phase transformation at 882°C to a body-centered cubic crystal structure (β phase) with a space group symmetry Im3m (229) [4]. Fig. 2.2 shows both crystal structures with lattice parameters and the most densely packed types of lattice planes [4]. The c/a ratio for pure titanium (α phase) is 1.587 smaller than the ideal ratio of 1.633 for the hexagonal close-packed crystal structure (hcp). This reduced c/a ratio makes the packing density of prism planes larger compared with the basal planes which could be favorable for slip on prism planes rather than on basal planes [5]. The lattice parameter value of pure β titanium is at 900°C and the close-packed directions are the four <111> directions [4]. Titanium α phase exhibits a high elastic anisotropy. The Young’s modulus in single crystals varies from 145 GPa with a stress axis parallel to the c-axis to 100 GPa with a stress axis perpendicular to the c-axis. The Young’s modulus of the β phase cannot be measured in pure titanium because this phase is not stable at room temperature. However, it is possible to retain the metastable β phase at room temperature using special alloying elements and very fast cooling rates [4].

Fig. 2.2 Crystal structure of hcp α (left) and bcc β (right) phase (adapted from ref. [5])
Alloying elements in titanium are classified as $\alpha$ or $\beta$ stabilizers. This designation depends on how those alloying elements affect the position of the $\alpha/\beta$ transformation temperature making one of the phases as predominant [4]. Fig. 2.3 shows schematic phase diagrams of common alloying elements for titanium. Aluminum is the most common $\alpha$ stabilizer due to its large solubility in $\alpha$ and $\beta$ phases. Commercially pure titanium alloys uses oxygen as an interstitial $\alpha$ stabilizer due to its good solubility and ability to improve the strength [4]. V, Mo and Nb in enough concentrations can fully stabilize the $\beta$ phase at room temperature and are very common in titanium alloys. Fe, Mn, Cr, Ni and Si are also typical in titanium alloys and partially stabilize the $\beta$ phase (i.e. $\beta$ eutectoid forming elements). Zr and Sn have complete solubilities in the $\alpha$ and $\beta$ phases of titanium and are neutral in titanium alloys, because they cause a very small change in the $\alpha/\beta$ transformation temperature [4]. However, in commercial alloys they are considered as $\alpha$ stabilizers because Zr is isomorphous with titanium (i.e., both elements have the $\beta$ to $\alpha$ allotropic phase transformation) and Sn behaves as a stabilizer when Al is also present [4].

![Phase diagrams of titanium alloys](image)

Fig. 2.3 Common alloying elements on phase diagrams of titanium alloys [4]
2.1.3 β/α Transformation

The transformation from bcc β phase to hexagonal α phase occurs when the most densely packed planes (110)_β becomes the basal planes (0001)_α of the hexagonal α phase as shown in Fig. 2.4 [5]. This transformation follows the called Burgers relationship where a bcc crystal can transform into one of 12 variants with different orientations. However, the distance between basal planes in α is slightly larger than the distance between the parent (110)_β planes. This generates a small atomic distortion which in turn reduces the c-axis relative to the a-axis in the hcp α phase (i.e. c/a ratio smaller than ideal) [5]. This transformation always follows the Burgers relationship, but the final stable phase can be affected by the alloying elements mentioned before and by the cooling rate, so the process can occur martensitically or with a controlled and slow nucleation and growth [4]. Martensite transformation occurs when the cooling rate is fast enough to impede a transformation under thermodynamic equilibrium (i.e. controlled and slow nucleation). It involves a movement of atoms by a shear type process resulting in a distorted hexagonal crystal lattice that is designated as α’ with an acicular morphology [4].

![Fig. 2.4 Schematic representation of (a) α phase (b) β phase and (c) Burgers relationship between α and β during transformation [6]](image-url)
In titanium alloys when the solute content reaches certain level, the hexagonal structure of the martensite becomes even more distorted than $\alpha'$. In this case, the hexagonal unit cell loses its symmetry and must be described as orthorhombic martensite $\alpha''$ [4]. In addition, during fast cooling rates the precipitation of omega ($\omega$) phase occurs when the solute content is high enough to be close to the lower limit for $\beta$-phase retention in most titanium systems [7, 8]. This $\omega$ phase has a trigonal symmetry in high solute content alloys and hexagonal symmetry in leaner alloys. However, the precipitation of this phase is in very fine particles (size 2-4 nm) and is accompanied by a very undesirable phenomenon due to embrittlement [4, 9].

Equilibrium nucleation and growth is when the cooling rate from the $\beta$ phase field into the ($\alpha + \beta$) phase field is slow enough to allow a diffusional process [4]. Nucleation of the $\alpha$ phase starts at the $\beta$ grain boundaries forming parallel layers or plates. All those plates that are parallel to each other, belong to the same variant of the Burgers relationship and they are called $\alpha$ colony [4]. In such colonies, the $\alpha$ plates or laths are separated by the retained $\beta$ matrix, these colonies grow in the interior of a $\beta$ grain until they meet another $\alpha$ colony growing in the opposite direction. In this sympathetic nucleation and growth process, the colony size is always smaller than the parent $\beta$ grain [4]. This microstructure is called basket weave or Widmanstätten.

### 2.1.4 Alloy Classification

A common classification of titanium alloys by specific applications and no structural features is used in industry [10]. For instance, commercially pure (CP) titanium grades as 1,2,3,4, where the grade indicates the oxygen content from 0.18 wt% to 0.4 wt%, Ti-Pd (grade 7 and 16), Ti-3Al-2.5V (also known as half Ti64), Ti-0.3Mo-0.8Ni (grade 12) and BETAC (grade 19 and 20) are considered as corrosion resistant alloys [4, 10]. Titanium alloys as Ti-
6Al-4V, Ti-5Al-2.5Sn (grade 6), Ti-6Al-6V-2Sn (Ti 6-6-2), Ti-10V-2Fe-3Al (Ti 10-2-3), Ti-15V-3Cr-3Sn-3Al (Ti 15-3), Ti-5Al-2Sn-4Mo-2Zr-4Cr (Ti-17), Ti-4Al-4Mo-2Sn (Ti550), Ti-8Al-1Mo-1V (Ti-8-1-1) are considered high strength alloys. Finally, Titanium alloys as IMI679, IMI685, IMI829, IMI834, TIMETAL 1100 and titanium aluminides are classified as high temperature alloys [10]. However, titanium alloys are more officially classified based on the phases that are predominant in their microstructure. This classification can be represented in a pseudo-binary β-isomorphous phase diagram (Fig. 2.5).

![Fig. 2.5 Pseudo-binary β isomorphous phase diagram [4]](image)

### 2.1.4.1 α and near α titanium alloys

This group of alloys have α phase as predominant phase, but with some small amounts of β phase (2-5 vol. %) stabilized by iron [4]. The presence of β phase in small amounts improves the hydrogen tolerance and controls the recrystallization of α grains upon annealing. Aluminum is the main alloying element apart from Zr, Sn and interstitials (O, C, and N). All
these elements can be expressed as the aluminum equivalent in Eq. 2-1 and if it is above 9 wt.%, it can cause detrimental precipitations (generally Ti$_3$X) [10, 11].

\[
\text{wt\%} = \text{Al} + \left(\frac{1}{3}\right)\text{Sn} + \left(\frac{1}{6}\right)\text{Zr} + 10(\text{O} + \text{C} + 2\text{N})
\]

Eq. 2-1

\(\alpha\) alloys possess a high rate of work hardening that restrict their formability. The poor heat treatment response makes this group of alloys to be easily welded and suitable for very low temperature applications [11]. Ti-5Al-2.5Sn is one of the most common \(\alpha\) alloys and is used extensively for the manufacturing of cryogenic storage vessels [10, 11]. Near \(\alpha\) alloys exhibit higher strength than \(\alpha\) alloys due to the formation of \(\alpha'\) martensitic phase. Additions of silicon are frequently used to improve strength the creep resistance [11].

2.1.4.2 \(\alpha + \beta\) titanium alloys

The group of \((\alpha + \beta)\) alloys is characterized by the presence of both phases, \(\alpha\) and \(\beta\). However, the \(\beta\) phase is present in a more significant manner than in near \(\alpha\) alloys [4]. Most of these titanium alloys have high strength and formability and are designed for temperatures below 400\(^\circ\) C. Fig. 2.5 indicates the higher \(\beta\)-stabilizer content required for these alloys and the Ms curve below which the alloy should be fast cooled down from \(\beta\) phase field to the \(\alpha + \beta\) phase field in order to obtain martensitic transformation. However, this higher \(\beta\)-stabilizer content does not exceed the aluminum equivalent value of 9 wt\% that is critical for the formation of Ti$_3$X [10]. Ti-6Al-4V is the distinguished alloy in this group due to the good balance on properties such as strength, ductility, and fatigue [4]. However, a more detailed description of this alloy is present in a later section. The microstructures of \((\alpha + \beta)\) alloys can be classified into three categories: fully lamellar, equiaxed and bi-modal [4].

The fully lamellar microstructure consists of \(\alpha\) laths in a \(\beta\) matrix due to cooling rates slow enough to be in thermodynamic equilibrium. However, the typical manner to obtain this
microstructure is by an annealing treatment (β recrystallization) in the β phase field. Fig. 2.6 shows the processing route for common industrial practice to obtain the fully lamellar microstructure [4]. Step I is homogenization in the β phase field which transforms the raw (α + β) alloy into a more workable and homogeneous microstructure. This step eliminates any segregated or textured microstructure from any previous process (e.g. ingot melting) [4, 12]. Forging or rolling are the deformation processes in step II. A common practice is to deform the alloy above the β-transus due to lower flow stress and then deform the alloy below the β-transus to avoid excessive β grain growth. The recrystallization step is usually 30-50⁰C above the β-transus to avoid β grain growth. The cooling rate in step III is the most important processing parameter because it determines α lath thickness, α colony size and the thickness of α layers at the β grain boundaries. In addition, this parameter determines the change from a α colony (i.e. Widmanstätten or basket weave) microstructure to a martensitic microstructure. The specific cooling rate value for this transition varies with alloying elements, but for the most common (α + β) alloys (e.g. Ti-6Al-4V), it is 1000⁰C/min [4]. The final step IV can be used for age hardening by precipitation of second phase particles at the right temperature. However, it is usually just a stress relieving treatment [4].

![Fig. 2.6 Processing route for fully lamellar microstructure [4]](image-url)
Fig. 2.7 shows a typical fully lamellar microstructure at different cooling rates. Images a and b show cooling rates slow enough to obtain the lamellar microstructure (α colony). However, image c exhibits non-parallel positioned α laths, corresponding more with randomly oriented laths of martensitic microstructure due to the high cooling rate [4]. In fully lamellar microstructures the slip length is controlled by the α colony size, so the α colony boundaries are acting as barriers. Dislocation motion can easily occur across the α/β interface because α/β phases have 2 parallel slip systems and 2 others that are off by only $10^\circ$ [4]. Therefore, important mechanical properties as yield strength, ductility, High Cycle Fatigue (HCF) and microcrack propagation resistance are inversely proportional to slip length (i.e. α colony size in this case). Individual α laths in the martensitic microstructure play the role of barriers for dislocation motion due to the different crystallographic orientation. It is important to mention that the trend for crack propagation has a variation when the crack size becomes larger (i.e. changing from microcracks to macrocracks). There is an additional factor to consider when two adjacent areas with different crystallographic orientations along the crack front can bifurcate the crack into two different slip planes. Those different slip planes come from different and adjacent α colonies or individual α laths depending on the microstructure (lamellar or martensitic) [4]. Therefore, the bifurcation magnitude is proportional to the size of the microstructure feature. The resulting trend is that macrocrack propagation resistance is proportional to α colony or individual α lath size (i.e. slip length) on each case. This specific behavior of macrocracks in contrast with microcracks is frequently called retarded term or crack front geometry and it has an important effect on Low Cycle Fatigue (LCF) [4]. Slip length and crack front geometry are the factors to evaluate HCF or LCF including the R-ratio that can make one or the other factor the dominant in crack propagation [4].
Fig. 2.7 Fully lamellar microstructure of Ti-6242 affected by different cooling rates in step III: a) 1° C/min b) 100° C/min c) 8000° C/min [4]

The bi-modal microstructure consists of equiaxed primary $\alpha_p$ grains surrounded by a lamellar microstructure of $\alpha$ laths in a $\beta$ matrix (Fig. 2.8 and 2.9). In the homogenization process (step I), the cooling rate determines the size of the $\alpha$ laths which in turn influences the equiaxed primary $\alpha_p$ grain size [4]. The deformation in step II has to introduce enough stored energy (dislocations) to help with the recrystallization process in step III that is performed below $\beta$-transus in the ($\alpha + \beta$) field. The temperature in step III determines the volume fraction of equiaxed primary $\alpha_p$ grains. Therefore, the cooling rate in step I and the temperature in step III are the most important processing parameters in bi-modal microstructures because they define the volume fraction and size of the equiaxed primary $\alpha_p$ grains which in turn define the $\beta$ grain size (i.e. distance between $\alpha_p$ grains) [4]. The temperature in step III (recrystallization) also determines a second parameter in bi-modal microstructures. This parameter is the alloy element partitioning which is proportional to the volume fraction of $\alpha_p$. This effect is basically a diffusion of $\alpha$-stabilizer elements (e.g. oxygen and aluminum) that moves from the lamellar region towards the equiaxed primary $\alpha_p$ grains [4].
This partitioning leads to a lower strength within the lamellar region in the bi-modal compared with the fully lamellar microstructure. Thus, properties such as yield strength, ductility, microcrack propagation resistance, HCF and LCF strength will be better for bi-modal microstructure than for a fully lamellar one [4]. This is true as long as the governing parameter is the $\alpha$ colony size (slip length) and not the alloy element partitioning. However, if the governing parameter is the alloy element partitioning promoted by high volume fraction of $\alpha_p$, the trend could be the opposite in some properties depending on the temperature of recrystallization that as mentioned before, determines the volume fraction of $\alpha_p$ [4]. Fig. 2.9 shows an example of bi-modal microstructure.
Fully equiaxed microstructure consists of equiaxed $\alpha$ grains and equilibrium volume of $\beta$ phase at the “triple points” of the $\alpha$ grains (Fig. 2.10). The processing route is in essence the same as for bi-modal microstructure (Fig. 2.11), but with a very slow cooling rate in step III (recrystallization). The slow cooling rate allows the formation and grow of $\alpha$ grains and no $\alpha$ lamellae. However, there is a second option to obtain fully equiaxed microstructure. In the recrystallization process (step III), the lower the temperature the higher the volume fraction of $\alpha$. Thus, fully equiaxed microstructure will be formed directly from the deformed lamellar microstructure.

Fig. 2.10 Optical micrograph of fully equiaxed microstructure of Ti-6242 alloy [4]

Fig. 2.11 Processing route for fully equiaxed microstructure of (a + b) alloys [4]
Important mechanical properties as yield strength, ductility and microcrack propagation resistance have the same trend than as fully lamellar. The $\alpha$ grain size in fully equiaxed microstructures plays the role of $\alpha$ colony size in fully lamellar. Therefore, the slip length and crack front geometry are the governing factors in fully equiaxed microstructures in a similar manner as for fully lamellar [4].

2.1.4.3 $\beta$ titanium alloys

As Fig. 2.5 shows these alloys do not transform into martensitic microstructure as the Ms curve ends on the border of $(\alpha + \beta)$ and $\beta$ metastable alloys. Metastable $\beta$ alloys are stronger that $(\alpha + \beta)$ alloys because they can be hardened to a higher yield stress by precipitating $\alpha$ particles. The processing route of Fig. 2.12 is to obtain the $\beta$-annealed microstructure which controls the continuous $\alpha$ layers at $\beta$ grain boundaries. The presence, absence or influence of the continuous $\alpha$ layers on mechanical properties is the key microstructural feature of all $\beta$ titanium alloys [4]. The Beta 21S alloy shows the $\beta$-annealed microstructure where the recrystallization temperature (step III) determines the $\beta$ grain size. The time and temperature of aging (step IV) determines the precipitation volume, size and distribution of a platelets in Fig. 2.13 [4].

Fig. 2.12 Processing for $\beta$ annealed microstructure of heavily stabilize $\beta$ titanium alloy [4]
2.1.5 Titanium Alloy Ti-6Al-4V

This alloy can be heat treated at different strength levels, it is weldable and easy to machine [10]. It has an extensive use in the aerospace industry and has the ($\alpha + \beta$) type of microstructures. For this reason, the versatility to control the microstructure and improve specific properties, makes this alloy ideal for a wide range of applications [14]. Ti-6Al-4V is the most widely used titanium alloy for structural aircraft components. Static and rotating parts in the gas turbine engines such as blades, airframes, fuselage, fan discs and housings are some examples [14]. In the biomedical sector, it is also used for joint replacement parts such as hip, jaw and knee implants. This alloy is commercially known as Grade 5 Titanium and the standard chemical composition is given in Table 2.1 [14]. As mentioned before, this alloy belongs to the ($\alpha + \beta$) type of microstructures (i.e. fully lamellar, bi-modal or fully equiaxed). Cooling rate and temperature in the processing route define the $\alpha$ colony and $\alpha$ grain size (i.e. slip length) in fully lamellar, bi-modal and fully equiaxed microstructure respectively. Therefore, the mechanical properties are dependent on microstructure and texture [14].
Table 2.1 Chemical composition of Ti-6Al-4V (ASTM B863 Grade 5 – UNS R56400) [14]

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>C</th>
<th>Fe</th>
<th>H</th>
<th>N</th>
<th>O</th>
<th>V</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5.5-6.75%</td>
<td>≤ 0.1%</td>
<td>≤ 0.4%</td>
<td>≤ 0.015</td>
<td>≤ 0.05%</td>
<td>≤ 0.2%</td>
<td>3.5-4.5%</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Wrought Ti-6Al-4V has a modulus of elasticity of ~114 GPa, the yield strength is 830 -1100 MPa and the elongation is 10 - 15%. For cast Ti-6Al-4V, the yield strength is 870 - 1050 MPa and the elongation is 8 - 10% [14]. For Ti-6Al-4V (stress relieved condition) produced via the LENS™ process (Laser engineered net-shape) an additive manufacturing technology, the modulus of elasticity is ~116 GPa, the yield strength is - 832 - 1065 MPa and the elongation is 0.8 - 4.9% [15]. For electron beam additive manufacturing process (EBAM®), the modulus of elasticity is 120.7 - 126.3 GPa, and the yield strength is 713.7 - 723.7 MPa [16].

2.2 Linear Friction Welding

2.2.1 Description of the Process

Linear friction welding LFW is a solid-state welding process of two workpieces under compressive forces [17, 18]. During the process, one workpiece is stationary while the other one is in motion, this friction generates heat that plasticizes the contact zone and a final forging pressure is applied to consolidate the joint (Fig. 2.14a) [19]. Vairis and Frost [20, 21] describe the process in four distinct phases. At phase I, the two workpieces are placed into contact under a certain pressure. The contact area is augmented with reduction of asperities and heat generation due to solid friction. In phase II, the heat generation is enough to increase the area of contact to 100% and expulsion of viscous material from the interface (i.e. initial flash formation). In the specific case of Ti-6Al-4V, this occurs when the interface reaches the β-transus temperature [22]. Phase III is the equilibrium phase where the flash formation is more
visible and the axial shortening is present at a constant rate [23]. Phase IV is known as deceleration and forging phase where in less than 0.1 seconds the two workpieces are brought to rest and a final forging pressure is applied to finish the joint.

![Diagram of Linear Friction Welding process and Integrated blisk](image)

**Figure 2.14.** (a) Diagram of Linear Friction Welding process, (b) Integrated blisk [24]

An important technical advantage of LFW over regular welding or any additive manufacturing technique is the solid state nature of the process. Therefore, all problems associated to solidification are avoided (e.g. porosity, hot cracking, segregation and so on). Dissimilar materials can be welded due to lower peak temperatures [24]. The process is quick in terms of weld time, and the severe deformation at the weld zone usually leads to a refined microstructure that offers better strength at the weld line compared to the parent material [24]. Some disadvantages include the high cost of equipment and tooling, and the process can be very noisy. For these reasons, this method is normally used to manufacture high added-value components such as bladed discs (blisk) for aeroengines (Fig. 2.14b) [23-26]. The main economic advantage of LFW resides in the fact that for aircraft structural components
oversized ingots are machined to get the final product, so a large amount of material is wasted. Smaller workpieces can be joined with LFW to produce a component, so less material is required as an initial step [23]. Linear Friction Welding has 8 processing parameters. Frequency, amplitude, applied force and burn-off (axial shortening) are considered as the most important ones. However, ramp-up time (time to reach steady-state for frequency and amplitude), oscillation decay time (time of decaying from steady-state to stop), forging force (the last force used to consolidate the joint after oscillation), and forging force time (time of the forcing force application) are the other four [23]. Frequency and amplitude are two processing parameters that can be treated as a single input parameter, called average rubbing velocity. Changing either frequency or amplitude, but keeping the rubbing velocity constant has no significant effect on Ti-6Al-4V linear friction welds, and it is defined in Eq. 2-2 [27].

\[ v_r = 4 \text{ (Amplitude} \times \text{frequency)} \]  \hspace{1cm} \text{Eq. 2-2}

Currently there are five major companies that manufacture LFW equipment: ACB An Aries Alliance Company (France), APCI, LLC a Subsidiary of Stupp Bros., Inc (Indiana, USA), KUKA Systems Machinery Industry Company (Augsburg, Germany), Manufacturing Technology, Inc (Indiana, USA) and Thompson Friction Welding (UK). LFW equipment from these companies has the capability to operate with precision of ±0.025 mm and 0.04° [23]. Rubbing velocity, applied force and axial shortening (determined by the workpiece geometry) are the principal controllers of the final component microstructure. In general terms, Fig. 2-15 describes the thermal effects of those processing parameters on Linear Friction Welding (LFW).
Increasing the rubbing velocity (higher input power), increases the temperature gradient and the maximum temperature at the weld interface (blue line compared to green). It can be seen from the graph that the distance from the weld interface above $\beta$-transus is very similar comparing 540 mm/s with 120 mm/s, so the influence of rubbing velocity is not a significant variable on the size of heat affected regions. Reducing the applied force from 125 to 40 MPa (pressure), increases the distance from the weld above the $\beta$-transus (black line compared with blue line) and the maximum temperature at the weld interface [23]. This can be explained by the fact that more pressure gives more heat at the interface, but also more axial shortening which in turn means more flash ejection leading to a faster heat dissipation [19]. Then, the heat coming out is greater than the heat generated. The in-plane width which means
the material’s width in the oscillation direction represents the geometric influence (related to the axial shortening). Reducing the contact area from 40 to 10 mm (comparing blue with red lines, Fig 2.15) leads to a lower maximum temperature at the weld interface and less distance from the weld interface above the β-transus. This is due to faster axial shortening (burn-off) which means faster heat dissipation [23]. In this research, the processing parameters of LFW are considered just in an indirect manner because the specimens were provided by Honeywell with no specification about them. In consequence, the microstructure is evaluated as it is given.

2.2.2 Microstructure Development on Ti-6Al-4V

Regardless of the processing parameters in linear friction welding of Ti-6Al-4V, the microstructure development from the weld interface has three distinct zones: Parent material (PM), thermo-mechanically affected zone (TMAZ) and the welded zone (WZ) [23]. Usually for linear friction welding of Ti-6Al-4V, the PM is a bi-modal microstructure (i.e. primary $\alpha_p$ grains surrounded by a lamellar microstructure of $\alpha$ laths in a $\beta$ matrix) (Fig. 2.16a), the TMAZ is a distorted bi-modal microstructure (Fig. 2.16b) and the WZ is the zone that undergoes dynamic recrystallization (reaching temperatures above $\beta$-transus) [17, 23, 28, 29]. Thus, the WZ is a refined martensitic $\alpha'$ (needle-like) microstructure (Fig. 2.17a), as reported in other research publications about LFW of Ti-6Al-4V where the cooling rate was also critical [18, 25, 28, 29]. However, other authors reported the presence of a Widmanstätten microstructure (Fig. 2.17b) with a very small $\alpha$ colony size [17, 19, 22, 23]. Strong transverse texture ($10\overline{1}0$) [$11\overline{2}0$] at the WZ was reported by Karadge et al. [25] and they used laboratory scale blocks of (26 mm x 13 mm 70 mm) representing disc and blade for LFW. At the TMAZ there was not any evidence of texture. On the other hand, full scale samples with a weld interface area about 12 times that of the laboratory scale were also used for LFW by Karadge et al. [25]. In this
large size the WZ was also strong transverse textured (10\(\overline{1}\)0) [11\(\overline{2}\)0], but the TMAZ exhibits alternating bands of transverse (11\(\overline{2}\)2) [11\(\overline{2}\)3] (Fig. 2.18). The reason of that difference in TMAZ textured due to scale size is still unknown [23]. On the other side, Romero et al. [19] reported random texture at WZ when using high pressures (9P) and the same transverse textured as indicated by Karadge et al. [25] when using lower pressures (P).

Figure 2.16. (a) Optical image of Parent Material (PM) [25] (b) SEM image of TMAZ [17]

Figure 2.17. (a) Backscattered image of Martensitic \(\alpha\)' (needle-like) [29] (b) SEM image of Widmanstätten microstructure [17]
2.2.3 Mechanical properties

Vickers’ hardness test in the WZ reports higher values (e.g. 422±11 [30], 425±10 [19], 398±3 [17]) than the parent material (PM) (e.g. 302±20 [30], 328±20 [19], 349±3 [17]) which is due to the refined microstructure in the WZ. The TMAZ has the same microstructure of parent material, but heavily distorted with no recrystallization evidence. Grujicic et al. [31] reported values on the TMAZ of 360-400 HV, which are higher than those on the parent material, showing the effect of strain hardening [30, 31]. Hirosi et al. showed the same trend of hardness and varying the applied force across the three zones in LFW [28] (Fig. 2.19). However, Wanjara et al. [17] reported hardness values at the TMAZ lower than the parent material. In addition, they showed that increasing frequency, amplitude, applied force (pressure) and axial shortening reduces the hardness at the TMAZ as shown in Fig. 2.20. These results are against others that indicate that increasing applied force or other input parameters will increase the cooling rate which in turn should increase hardness [27, 28].

Figure 2.18. (a) Geometry and directions (b) Crystallite orientation of WZ and TMAZ of full scale samples (adapted from [25])
Figure 2.19. Distribution of hardness across LFW [28]

Figure 2.20. Vickers hardness across LFW line with the effect of some processing parameters a) frequency b) applied force c) amplitude and d) axial shortening [17].
The difference in these hardness results respect to the TMAZ compared with the PM indicates that more research has to be done on the influence of processing parameters. However, this is not the focus of this thesis. Different tensile tests have been performed to evaluate tensile properties of LFW [17, 28, 30, 32]. If the welded zone does not present any contaminants [17, 27, 33] or insufficient plasticization [32] due to processing parameters (e.g. small friction force), the tensile test will fail in the parent material. However, Wanjara et al. [17] showed tensile test failure at the TMAZ, in agreement with the lower hardness values of the TMAZ compared with PM mentioned before. This research section is specifically focused into the individual response of LFW zones to tensile test, so a special test is designed to make the specimens to fail on each specific LFW zone.

2.3 Electron Beam Additive Manufacturing

2.3.1 Description of the process

Electron Beam Additive manufacturing (EBAM®) is a metal additive manufacturing technology that uses an electron beam as heat source, wire as feedstock and a vacuum chamber for the electron beam that also protects the alloy from oxygen reaction [34]. This technique can produce large scale metal structures of 19 ft x 4 ft x 4 ft (5.79 m x 1.22 m x 1.22 m) of titanium alloys in days. Several months are required to produce same large-scale metal structures via casting and forging with more material waste. In addition EBAM® works not only for rapid prototyping and part production, but also for repairing and remanufacturing applications [34]. Figure 2.21 shows a schematic of EBAM® by Sciaky Inc. The technology also offers a dual wirefeed system to combine two different metal alloys into one melt pool to create gradients or a custom alloy.
2.3.2 Microstructure Development on Ti-6Al-4V

In general terms an additive manufacturing technique produces a columnar microstructure due to the layer by layer deposition. The YZ plane in Figure 2.22 shows two distinct microstructural features labeled as zone $A$ and $B$. The elongated grains strongly oriented in the $z$-direction of zone $A$ are consistent with the greatest gradient of temperature associated with the maximum heat extraction rate in the $z$-direction during additive manufacturing [35, 36]. Hayes et al. [16] reported AM-$\beta$ annealed with very little variation in $\alpha$-lath thickness due to the uniform and strong epitaxial growth from bottom to top (Fig. 2.23a,b). Zone $B$ comprises of grains more oriented in the $y$-direction. They have a pronounced variation in $\alpha$-lath thickness and a more scattered orientation due to the competing growth from the side wall of the molten pool (Fig. 2.23c,d) [16]. Several tensile tests were already reported on those microstructures to assess their influence on tensile mechanical properties [16]. Therefore, the interest of this research is in mechanical properties from a fatigue test on those two types of microstructures.
Figure 2.22. YZ Cross-section of an ELI Ti-6Al-4V build [16]

Figure 2.23 Backscatter electron micrographs of Zone A (ab) Zone B (c,d) [16]
2.3.3 Mechanical properties

Samples extracted from zone A and B were tested by Hayes et al. [16] in uniaxial tension in the z-direction to evaluate the individual microstructural features. Figure 2.24 shows the tensile test of several specimens taken from zone A and B. Zone B exhibits a wider variation in the yield strength compared with zone A. Hayes et al. determined that such yield strength variation is due to the greater thickness variation and more scattered orientation of α-laths of zone B [16]. Table 2.2 has specific reported data for the yield strength of zone A and B.

![Subscale Tensile Data Zones A and B](image)

Figure 2.24. Tensile test results of specimens taken from zone A and B (adapted from [16])

<table>
<thead>
<tr>
<th></th>
<th>Zone A: Yield Strength</th>
<th>Zone B: Yield Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>713.7 MPa</td>
<td>723.7 MPa</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>27.11 MPa</td>
<td>59.10 MPa</td>
</tr>
</tbody>
</table>

Table 2.2 Statistical distribution of yield strength in EBAM® Ti-6Al-4V [16]
2.4 Ultrasonic Fatigue Testing

2.4.1 Characteristics of ultrasonic fatigue

Conventional fatigue approach provides useful information of mechanical properties. However, several applications of Ti-alloys (e.g. Ti-6Al-4V) are required for safe operation over long periods of time, extending $10^9$ cycles [37]. Conventional fatigue tests as electromagnetic shakers or servo-hydraulic systems can achieve $10^9$ cycles in weeks, so a single S-N curve would last months [38]. This approach is impractical to understand the material behavior at this regime. Therefore, ultrasonic fatigue testing offers an alternative where $10^9$ cycles can be reached in less than a day. An ultrasonic fatigue system contains a generator, transducer (piezoelectric elements that generate the mechanical movement), booster (typically acting as an amplifier), acoustic horn (applies the vibration energy to the specimen) and specimen (Fig. 2.25). Each part of the system has to satisfy the resonance condition or natural frequency [39].

![Ultrasonic fatigue testing machine diagram](image)

Figure 2.25 Ultrasonic fatigue testing machine. 1-generator, 2- transducer, 3-booster, 4-horn, 5-specimen. $u(x)$ displacement, $\sigma(x)$ stress [40].
The resonance system design is made with specific dimensions to offer a mode shape with maximum amplitude deformation on the specimen and not on any other part of the system [39, 41]. Usually 20 kHz ± 500 Hz is the eigenfrequency used to perform fatigue tests. In ultrasonic fatigue testing a sound wave is transmitted from the piezoelectric material to the specimen, under resonance conditions to cause vibration on the specimen [42]. In 1950 the first ultrasonic fatigue test at 20 kHz was performed [43]. Later, authors proposed higher frequencies such as 199 kHz, but those too high frequencies generate excess of heat and the geometrical dimensions are too small making the test impractical [43]. Consequently, the working design at 20 kHz is the basis of the actual research in ultrasonic testing machines.

Cylindrical dogbone shape samples have been used as prototypes for several tension-compression ultrasonic fatigue test machines [38, 39, 43-47] at elevated temperatures [42, 48] or using the high cost but commercially available gigacycle ultrasonic fatigue testing system SHIMADZU USF-2000 [49, 50]. In addition, different ultrasonic fatigue prototypes for torsional [51, 52] and multiaxial state [51, 53] have been successfully developed. However, all these previous studies are not focused in ultrasonic bending test on cantilevers for individual microstructural feature evaluation. Only Wilkinson and Gong [41] tried ultrasonic fatigue test on micro-cantilevers with a hammer shape to increase the inertia and reach the stress level they wanted to evaluate (Fig. 2.26). Nevertheless, there is not a detailed description of the resonance system, oscillation measurement and how the failure of the first cantilever will affect the resonance frequency of the system which in turn changes the stress state on other cantilevers.
Our approach here is again to evaluate a selective microstructure of an EBAM®-Ti-6Al-4V process (zone A and B) as for the conventional fatigue case ($10^7$ cycles), but in the regime of very high cycles ($10^9$). For this purpose, we will use an ultrasonic welding machine manufactured by Branson Ultrasonics with certain modifications to perform the fatigue test. The pre-selection of booster, horn, specimen shape and dimensions will be based on simulations of the process by COMSOL Multiphysics®.

### 2.4.2 The design of the ultrasonic fatigue system

The test specimen needs to oscillate enough to obtain the stress level required to break and construct the $S$-$N$ curve for the fatigue analysis. To introduce a maximum amplitude of oscillation, the cantilever should be excited at its resonance frequency. Geometry and material parameters (e.g. dimensions, elastic modulus $E$ and density $\rho$) are the input information needed to design the entire resonance system that includes actuator (piezoelectric), booster (amplifier), horn (vibration energy transmitter) and specimen. COMSOL Multiphysics® is a commercial...
A package designed to solve engineering problems applying the finite element method. A more detailed description of Finite Element Analysis (FEA) can be found in [54-57]. In general terms, it is difficult to determine if a realistic model is well-posed or not to get convergence to a unique solution in a FEA software. Therefore, some simplifications are usually implemented to understand and estimate the behavior of a specific phenomenon [36]. The focus of this work is first to find the resonance frequency (eigenfrequency) of the system around 20 kHz that provides the mode shape (cantilever oscillation) required. A trial and error method is performed to find the shape and dimensions of the specimen in order to have a balance between the specimen size requirements due to individual microstructure evaluation and mode shape of the eigenfrequency. Once the first simulation provides the eigenfrequency of the system at the right mode shape, a second simulation studying a range of frequencies around the eigenfrequency is set [40]. The electric potential (V) varies to evaluate the stress and deflection of the cantilever matching lower values of 460 MPa and higher values of 980 MPa. Those two limit values are common in an S-N curve of Ti-6Al-4V [37, 49]. For these two simulations COMSOL Multiphysics® has two interfaces (i.e. physics), Solid Mechanics (solid) under Structural Mechanics branch and Electrostatic (es) under the AC/DC branch.

2.4.2.1 The solid mechanics interface

This interface usually has the purpose to determine the strength of a structure, in order to understand the limitations to avoid accidents. However, it is also useful to determine dynamic properties, such as time-dependent loads and for the purpose of this work natural frequencies [58]. In general terms, a structure can be statically determinate or statically indeterminate. For the determinate case, all forces in a system can be calculated just by equilibrium considerations. However, in real life, indeterminacy is more common due to the internal stress distribution calculation when it is required. In other words, deformations must
be taken into account to calculate the acting forces in a statically indeterminate system. Most of structural mechanics analysis are based on three type of equations. Those equations are equilibrium, compatibility and constitutive. The equilibrium equations are based on Newton’s second law, where the sum of all forces acting on a body for structure equilibrium are zero. The internal forces are the stresses that in three dimensions can be represented by the stress tensor (Eq. 2-3).

\[
\sigma = \begin{bmatrix}
\sigma_{xx} & \sigma_{xy} & \sigma_{xz} \\
\sigma_{yx} & \sigma_{yy} & \sigma_{yz} \\
\sigma_{zx} & \sigma_{zy} & \sigma_{zz}
\end{bmatrix} \tag{2-3}
\]

Each element in the matrix represents a stress component on a unit area in the material. The first index is the force component direction and the second index is the normal to the surface where the force acts [59]. The solid mechanics interface of the software reformulates Newton’s second law in terms of the stresses (Eq. 2-4).

\[
\nabla \cdot \sigma + f = \rho \frac{\partial^2 u}{\partial t^2} \tag{2-4}
\]

In this equation \( \rho \) is the mass density, \( u \) is the displacement vector and \( f \) is the external force [58]. The compatibility equations are required to take deformation into account. Strain represents relative deformation inside the material. In a simple elongation of a bar (Fig. 2.27), the engineering strain, \( \varepsilon \), is the ratio of displacement \( \Delta \) and the original length, \( L_0 \) [58].

![Figure 2.27 Engineering strain definition for pure extension](image)
The strain in three dimensions, as the stress, can be represented by a tensor (Eq. 2-5). All elements in the strain tensor matrix have specific spatial distribution, since they are derived from a displacement field. Specific geometric relations are the compatibility conditions in the continuum level.

\[
\varepsilon = \begin{bmatrix}
\varepsilon_{xx} & \varepsilon_{xy} & \varepsilon_{xz} \\
\varepsilon_{yx} & \varepsilon_{yy} & \varepsilon_{yz} \\
\varepsilon_{zx} & \varepsilon_{zy} & \varepsilon_{zz}
\end{bmatrix}
\]  
Eq. 2-5

Finally, the constitutive equations include the material properties factor. A material model establishes the connection between stress and strain. In contrast, with the previous equations, constitutive relations are phenomenological and not from first principles. The most common material model is linear elasticity, also known as Hooke’s law. In this model, the stresses are proportional to strains and the modulus of elasticity \( E \) is that proportionality. The bar in tension of figure 2.27 also shrinks in the transverse direction, so the relation between the strain in the axial and transverse direction is given by the Poisson’s ratio \( \nu \) (Eq. 2-6). The 3D visualization of Hook’s law is in Eq. 2-7, where \( D \) is a symmetric 6 x 6 matrix that in the isotropic case is only a function of \( E \) and \( \nu \) [58]. This COMSOL interface has many material model families (e.g. Elastoplastic, Creep, Hyperelastic, etc.), but for an isotropic linear elastic solid, it formulates a combined system with the Navier’s equation (Eq. 2-8).

\[
\varepsilon_{yy} = \varepsilon_{zz} = -\nu \varepsilon_{xx}
\]  
Eq. 2-6

\[
\begin{bmatrix}
\sigma_{xx} \\
\sigma_{yy} \\
\sigma_{zz} \\
\sigma_{xy} \\
\sigma_{yz} \\
\sigma_{xz}
\end{bmatrix}
= \begin{bmatrix}
\varepsilon_{xx} \\
\varepsilon_{yy} \\
\varepsilon_{zz} \\
\varepsilon_{xy} \\
\varepsilon_{yz} \\
\varepsilon_{xz}
\end{bmatrix}
D
\]  
Eq. 2-7

\[
\frac{E}{2(1+\nu)} \left( \frac{1}{(1-2\nu)} \nabla(\nabla \cdot \mathbf{u}) + \nabla^2 \mathbf{u} \right) + f = \rho \frac{\partial^2 \mathbf{u}}{\partial t^2}
\]  
Eq. 2-8
2.4.2.2 The Electrostatic theory interface

This interface studies non-moving electric charges that creates an electric field. The equation that relates the electric field with the charge density is known as Gauss’s law (Eq. 2-9) and it appears in this interface in the differential form. The left term of the equation is the divergence of the electric field, which represents the volume density of the inward ($<$ 0) or outward ($>$ 0) electric field vector. $\rho$ represents the charge density and $\varepsilon_0$ is the electric constant that represents the capability of the vacuum to permit electric field lines, also called vacuum permittivity or permittivity of free space [60].

$$\nabla \cdot \vec{E} = \frac{\rho}{\varepsilon_0}$$

Eq. 2-9

Electrostatic theory resides on the assumption of an electric field irrotational (i.e. curl free). It means that the electric field does not have the tendency to rotate, which can be expressed with Maxwell’s equation (Eq. 2-10) or with the static version of Faraday’s law (Eq. 2-11). In other words, the magnetic field must not change with time [60]. Therefore, the electric field can also be expressed as the gradient of a scalar potential (voltage $V$) (Eq. 2-12).

$$\nabla \times \vec{E} = 0$$

Eq. 2-10

$$\frac{\partial \vec{B}}{\partial t} = 0$$

Eq. 2-11

$$\vec{E} = -\nabla V$$

Eq. 2-12

The combination of Eqs. 2-9 and 2-12 provides a useful relation in engineering applications except to represent dielectric materials, in which charges are not free to move compared with negative charges (electrons) in conductive materials [60]. However, bound charges (positive and negative) in dielectric materials can be displaced by the external electric field and form induced electric dipoles. This polarization effect changes the electric field, then
Eq. 2-9 is modified into Eq. 2-13. Furthermore, COMSOL Multiphysics® uses the definition of the electric displacement field $D$ to fully describe the electrostatic phenomena under this interface (Eq. 2-14) [60].

$$\nabla \cdot (\varepsilon_0 \vec{E} + \vec{P}) = \rho$$ \hspace{1cm} \text{Eq. 2-13}

$$\nabla \cdot D = \rho$$ \hspace{1cm} \text{Eq. 2-14}

### 2.4.2.3 Piezoelectric effect multiphysics coupling node

This coupling node is an automatic available connection made by the software in the multiphysics node. It passes the material properties from the solid mechanics interface to the charge conservation node in electrostatic interface. The charge conservation node implements the electrostatic equations and does not require any additional setting when it is coupled with the multiphysics node [61]. The piezoelectric effect is present in some solid materials when an electric dipole moment occurs. Lead zirconate titanate (PZT) are the most widely used piezoelectric ceramics. When there is a mechanical stress on the material, a variation in the positive and negative charge centers occurs, which results in an external electric field (Fig. 2.28) [62].

![Figure 2.28](image)

Figure 2.28 (a) Ideal perovskite structure; (b) Distorted and polarized structure (adapted from [62])
The piezoelectric effect can also occur in the reverse direction. If an electric field is applied to the piezoelectric material, it will exhibit a structural distortion. The structure contracts and expands as a consequence of the electric field, releasing the energy in the form of a sound wave. Figure 2.29 shows sound wave generation with the inverse piezoelectric effect [63].

![Sound wave generation](image)

Figure 2.29 Inverse piezoelectric effect [63]

### 2.4.2.4 Eigenfrequency analysis

An elastic structure is prone to vibrate with a maximum amplitude at certain frequency, called natural frequency, eigenfrequency or resonance [58]. If the structure deforms elastically in the corresponding eigenfrequency, it is called the eigenmode (see example in Fig. 2.30). The eigenmode is only the shape of the mode and it does not provide any information about any physical vibration. The real deformation, stress or any physical information can only be determined by an actual excitation of the material [64]. The expressions for the eigenfrequency of a cantilever beam in terms of stiffness, mass and geometry are Eqs. 2-15 and 2-16 [64, 65].
Each case is different depending on those variables. For the cantilever beam, \( m_{\text{eff}} \) is the effective mass, \( E \) is the Young’s modulus, \( I \) is the area moment of inertia and \( l_{\text{cl}} \) is the length of the cantilever [65]. The combination of inertia and linear elasticity, through Newton’s second law and with appropriate boundary conditions, the resulting system of equations in the solid mechanics interface resolves for eigenfrequencies in a given system [58]. The determination of the eigenfrequency is very important in structural engineering. It can predict an excessive stress or noise emission due to a periodic excitation that is in resonance with the structure. For the purpose of this work, it provides the shape and dimensions of specimens that are at the right mode shape (eigenmode) for the ultrasonic fatigue study.

\[
\begin{align*}
f_{\text{res}} &= \frac{1}{2\pi} \cdot \sqrt{\frac{k}{m_{\text{eff}}}} \\
k &= \frac{3 \cdot E I}{l_{\text{cl}}^3}
\end{align*}
\]

Eq. 2-15

Eq. 2-16

Figure 2.30 The first two eigenmodes (mode shape) of a cantilever beam [58]

2.5 Conventional Fatigue Testing

2.5.1 Fatigue of metals

Fatigue is defined as a localized and progressive damage accumulation in a material during cyclic tensile-compression stresses below its yield strength [14]. The focus of this
section is not to provide an exhaustive description of fatigue phenomena, but to explain some
general concepts about fatigue and specifically the fatigue method on EBAM®-Ti-6Al-4V
specimens used in this thesis. First, description and differences of high and low cycle fatigue
behavior will be discussed. Then, some basic concepts about fatigue fracture mechanics will
be described. Finally, a special four-point fatigue bending test will be described in detail and
the reasons for its selection on this research will be explained.

2.5.2 Low and high cycle fatigue

The Wöhler or S-N diagrams represent the stress amplitude (σₘᵢₓ) vs the number of load
cycles on a material under test. These diagrams are used to design metallic components in order
to resist a given number of cycles. The conventional fatigue testing machines are hydraulic and
electric with a maximum frequency of 100 Hz [43]. Low Cycle Fatigue (LCF) regime
corresponds to less than 10⁵ cycles (Nᵣ < 10⁵ cycles) and High Cycle Fatigue (HCF)
corresponds to 10⁵ < Nᵣ < 10⁸ cycles [14, 37, 43]. The cyclic load in LCF is below, but close
to the yield strength of the material, but the presence of defects such as notches or scratches at
the surface act as stress concentrators. The local plasticity at those defects creates the
conditions for crack initiation. Normally, a fatigue test in a laboratory uses a sinusoidal wave
for the maximum and minimum stress (Fig. 2.31). From Eqs. 2-17 to 2-20, the ratio R is defined
as the minimum stress (σₘᵢₙ) divided by the maximum stress (σₘᵙ), which can be zero (when
the minimum stress is zero), positive (tension-tension test) or negative (tension-compression
test). The mean stress (σₘ) and the stress range (Δσ) are also used to describe a stress cycle
[66]. The definition of the different fatigue stress variables is:

\[
\sigma_m = \frac{\sigma_{max} + \sigma_{min}}{2}
\]  \hspace{1cm} \text{Eq. 2-17}

\[
\sigma_a = \frac{\sigma_{max} - \sigma_{min}}{2}
\]  \hspace{1cm} \text{Eq. 2-18}
\[ \Delta \sigma = \sigma_{\text{max}} - \sigma_{\text{min}} \]  

\[ R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} \text{ or } \frac{K_{\text{min}}}{K_{\text{max}}} \]  

**Figure 2.31.** Common variables used for fatigue prediction [66]

**2.5.3 Fatigue fracture mechanics**

The presence of defects at the surface as notches or scratches and internal defects as inclusions or pores exhibits localized plasticity under cyclic stress application and this is the cause of crack initiation. Localized plasticity occurs by dislocation slip, and the accumulation of slip steps creates the so-called persistent slip bands (see Fig. 2.32) [14]. After crack initiation there is a stage of crack growth. This stage is described by the stress intensity factor \( \Delta K \), a concept from linear elastic materials which assumes that the plastic zone at the crack tip is small enough to be negligible. In general terms, the stress intensity factor can be considered as a prediction of how much amplification of the applied stress is caused by the present crack. The magnitude of this factor depends on size and location of the crack, sample geometry and
the magnitude and distribution of the applied stress on the material. For a specific crack length \( a \) and an applied stress range \( \Delta \sigma \), the stress intensity factor \( \Delta K \) can be calculated from Eq. 2-21 [14].

\[
\Delta K = Y \Delta \sigma \sqrt{\pi a}
\]

Eq. 2-21

\( Y \) is the geometric factor that depends on crack and sample size and shape. From equation 2-21 it can be seen that the stress intensity factor \( \Delta K \) is proportional to the applied stress range \( \Delta \sigma \).

Paris and Erdogan [67] introduced the concept that the rate of crack growth depends on the stress intensity factor. The crack growth rate \( da/dN \), in which \( a \) denotes the crack length and \( N \) denotes the number of cycles, can be plotted as a function of \( \Delta K \) (Fig. 2.33) [68].

Figure 2.32 Schematic geometry of persistent slip bands (adapted from [69])
Figure 2.33 Crack growth rate \(\frac{da}{dN}\) vs stress intensity factor range \(\Delta K\) (adapted from [70])

In Region 1, \(\Delta K_{th}\) represents the stress intensity factor threshold, which means that below this value cracks do not propagate. Region 3 shows a continuous increment of crack growth rate, until reaching a limit where fracture occurs. At this limit of fracture, the stress intensity factor \(K_{max} (\Delta K = K_{max} - K_{min})\) is known as fracture toughness \(K_c\). In contrast, the most interesting region, 2, shows a constant crack growth rate. The linear relationship in Region 2 is represented by Eq. 2-22 and is known as Paris-Erdogan law [14, 67], where \(C\) and \(m\) are constants that depend on the material [14].

\[
\frac{da}{dN} = C (\Delta K)^m
\]

Eq. 2-22
The $R$ ratio (Eq. 2-20) plays an important role in crack growth rate ($da/dN$) and stress intensity factor threshold ($\Delta K_{th}$) because of crack closure. Crack closure is literally when the crack closes during unloading and the concept was initially given by Elber [71]. There is a stress level at which a crack opens ($\sigma_{op}$) and can be used to calculate the effective stress intensity factor range ($\Delta K_{eff}$) [14]. At high $R$ ratios, the crack is always open and $\Delta K$ is equivalent to $\Delta K_{eff}$ (see Fig. 2.34), but at low $R$ ratios, the crack closes and $\Delta K_{eff} < \Delta K$ [14, 71, 72]. Therefore, low $R$ ratios lead to slower crack growth rates and high $R$ ratios decrease the $\Delta K_{th}$. However, this phenomena is by far more complex and there are several proposal models to explain the influence of crack closure on crack growth behavior [72, 73].

![Figure 2.34 Schematic view of the effective and applied stress intensity factor range](image)

### 2.5.4 Fatigue on Ti-6Al-4V microstructures

The influence of the different types of microstructures of Ti-6Al-4V (i.e. bi-modal, lamellar and equiaxed) on fatigue properties is difficult to investigate in a systematic manner. Different microstructural parameters of each type of microstructure such as $\alpha_p$ content and size
in bi-modal, $\alpha$ grain size in fully equiaxed and $\alpha$ colony size or $\alpha$ lath thickness in fully lamellar have been used to compare the fatigue properties in literature [4, 49, 74, 75]. Zuo et al. [49] reported bi-modal with higher HCF strength than lamellar, but Nalla et al. [74] reported the opposite. However, Wu et al. [76] collected information from 75 sets of data in 21 references to observe the trend and comparison between the three type of microstructures and HCF strength. They found that the three type of Ti-6Al-4V microstructures have an important influence on fatigue properties. Bi-modal has the best fatigue properties, followed by lamellar and then equiaxed (see Fig. 2.35 with fitted exponential curves).

![Figure 2.35 Comparison of microstructure influence on fatigue properties [76]](image)

In lamellar microstructures, the fatigue cracks nucleate within slip bands or at the intersection of these slip bands with the adjacent $\alpha$ colony boundary. For lamellar microstructures formed at very high cooling rates (individual $\alpha$ laths), the fatigue cracks
usually nucleate at the longest and widest \( \alpha \) lath due to preferred slip band activity and only occasionally at the continuous \( \alpha \) layers at the \( \beta \) grain boundaries [4]. Fatigue cracks in bi-modal microstructure usually nucleates at the lamellar region with the same characteristics of the fully lamellar microstructure [4]. For fully equiaxed microstructures, crack nucleation occurs at the interconnection of \( \alpha_p \) grains. However, in order to understand fatigue behavior in these microstructures, the crack growth rate should be also analyzed. Crack propagation in lamellar Ti-6Al-4V is lower than in fully equiaxed due to the \( \alpha \) colony boundaries acting as stronger barriers than the \( \alpha_p \) grain interconnections in equiaxed microstructures. In contrast, equiaxed microstructures are less sensitive than lamellar to crack initiation. Therefore, bi-modal microstructures show a behavior that is in the middle of those two. For these reasons, bi-modal offers an overall better fatigue performance because it combines both resistance to crack initiation and propagation [14, 76].

2.5.5 Four-point bending fatigue test

Tensile properties are important to assess any material, but fatigue properties should be also considered. There is a wide spectrum of types of tests, depending on specimen size. Evaluating fatigue properties on specific microstructural features is difficult due to the small size of those features (e.g. LFW-Ti-6Al-4V zones or EBAM\textsuperscript{®}-Ti-6Al-4V zones). Four-point bending fatigue tests are ideal for small specimens and when full force reversal is not required. There are already a few ASTM standards to characterize flexure properties [77, 78], but they focus on non-metallic materials. Zhai et al. [79] describe the application of a four-point bending fatigue machine to test an 8090 Al-Li alloy by loading in one direction \( (R = 0.1) \) (Fig. 2.36). This method offers several advantages for the fatigue analyses of the EBAM\textsuperscript{®}-Ti-6Al-4V specimens. This fatigue test typically works with rectangular beams that produce a uniform
maximum stress on the surface, depending on the distance between inner rollers. Easy sample mounting and dismounting as no special gripping is required. It is also suitable to evaluate specific microstructures from small samples [79].

Figure 2.36 Four-point bend specimen geometry and the loading states [79].

Four-point bending fatigue test is susceptible to sample geometry. Therefore, specific dimensions should be used to standardized the test and eliminate the geometric influence. Zhai et al. [79] performed an analysis of thickness and load-span effect on stress distribution within the load-span. From beam theory the stress distribution along the load-span length is uniform and in pure-bending state (Fig. 2.36). The nominal maximum stress ($\sigma_{\text{nom}}$) in the load-span length can be calculated by Eq. 2-22. In Figure 2.37 where ($x/t$) values of 0 and 1 represent the position of the two loading rollers, Zhai et al. [79] using a finite element technique reported the ($t/h$) effect on stress distribution by varying the thickness ($h$) from 2 to 12 mm and remaining constant the load-span value of $t = 6$ mm. From these results, it is evident that the stress distribution is not uniform for all ratios except for $t/h = 1.33$, where the value is about 620 MPa which is just 3.3% greater than the calculated by Eq. 2-22 (600 MPa). They concluded the $t/h$ value between 1.2 and 1.5 offering the minimum variation in the stress distribution [79].

$$\sigma_{\text{nom}} = \frac{3P(L-t)}{wh^2} \quad \text{Eq. 2-22}$$
Figure 2.37 Surface stress distribution corresponding to different $t/h$ ratios [79]

The same finite element model was used to investigate the ratio of support-span/load-span ($L/t$) on the stress distribution. They set $L = 30$ mm, $h = 5$ mm and $t$ varying from 4 to 15 mm. The stress distribution is plotted in Figure 2.38. Ratios of ($L/t$) between 4 and 5 offer the minimum fluctuation of stress across the pure bending section ($\sim 5$ MPa fluctuation amplitude). The above results indicate that geometry is a factor that plays a role in the stress distribution on four-point bending test. However, ratios of $t/h = 1.2-1.5$ and $L/t = 4-5$ lead to the optimum testing geometry with uniform values and with a reasonable agreement with beam theory [79].

Figure 2.38 Surface stress distribution corresponding to different $L/t$ ratios [79]
CHAPTER 3. EXPERIMENTAL AND COMPUTATIONAL METHODS

3.1 Linear Friction Welding Specimens Preparation and Tensile Test

Linear Friction Welding (LFW) blocks of Ti-6Al-4V were provided by Honeywell with serial number 99193-R704 Research SN46. These samples have 59 mm in length, 70 mm in height (each block 31 mm in height) and 31 mm in width (Fig. 3.1). Dog-bone shape specimens were designed to be extracted via electron discharge machining (EDM) according to material availability, size of the interested region and tensile test equipment. Figure 3.2 shows the dimensions and shape of the dog-bone specimens. It also shows the EDM position to locate each individual LFW zone (i.e. welded zone (WZ), thermo-mechanically affected zone (TMAZ) and parent material (PM)) in the middle of the specimens to capture the tensile properties individually. The initial thickness of each dog-bone specimen was 1 mm and they were all reduced to 0.7 mm using 600-800 grit wet/dry SiC abrasive papers followed by polishing using a 0.04 μm colloidal silica suspension to remove the EDM recast layer from both sides. The edges were manually polished to mirror finish and to remove any remnant brown tint from EDM. Following preparation, all 24 specimens (8 per zone) were cleaned using a solution sequence of acetone, water-surfactant mixture, water and methanol. They were also etched with Kroll’s reagent to make the microstructure visible for optical analysis before and after the tensile test in a Leco Lx-31. However, some preliminary tensile tests suffered fracture in the PM region as shown in Figure 3.3. Therefore, the dog-bone shape specimens were modified based on yield strength estimations from microhardness measurements [17].
Figure 3.1 LFW blocks provided by Honeywell.

Figure 3.2 Dog-bone shape specimens extraction via EDM to capture the three LFW zones.
Figure 3.3 Backscatter images of dog-bone specimens showing fracture in PM (a) before and (b) after the test.

The hardness across the weld region was measured with a Vickers microhardness tester with a software for automated hardness values determination and a load of 100g. Five measurements per each zone were obtained. Table 3.1 shows averaged hardness values and the yield strength approximation, using the relation $H_v = 3\sigma_y$ [17]. Based on these preliminary results, the PM yield point is 83% of that of the WZ, then an area reduction of 75% is enough to make the WZ to yield first under tensile test. The current gage area is 1.4 mm$^2$ (0.7 mm x 2 mm). Therefore, reducing the width from 2 mm to 1.5 mm gives an area of 1.05 mm$^2$ (75% of the original area). Knowing the yield point of WZ and the force required to reach that yield point with the area reduction (i.e. 1324 MPa = $F/1.05$ mm$^2$ then $F= 1390$ N), the PM stress at that moment is 993 MPa (1390N/1.4 mm$^2$) which is 105 MPa lower than 1098 MPa. In addition, when the force reaches 1537 N (force required to reach the PM yield point), the stress on WZ is 1464 MPa. It represents 140 MPa above the WZ yield point and enough to guide the tensile test on evaluation of each specific LFW zone.
Table 3.1 Measured hardness and estimated yield strength values

<table>
<thead>
<tr>
<th></th>
<th>WZ</th>
<th>TMAZ</th>
<th>PM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hv</td>
<td>405 ± 1</td>
<td>367 ± 5</td>
<td>335 ± 5</td>
</tr>
<tr>
<td>σy</td>
<td>1324 MPa</td>
<td>1196 MPa</td>
<td>1098 MPa</td>
</tr>
</tbody>
</table>

Examples of the modified dog-bone shape specimens are shown in Figure 3.4. An EDM wire with a diameter of 165 μm (0.0065") was used to reduce the width from 2 mm to 1.5 mm (i.e. 250 μm on each side). Optical inspection on all specimens was performed to detect sharp edges or potential crack initiations. The edges were gently chamfered by manual polishing and cleaned again with the same chemical solution sequence mentioned before. The optical image in Figure 3.3a shows the microstructure of the specimen including the edges, indicating that no EDM recast layer is affecting the test. The tensile test equipment used in this work is a ZwickLine Z2.5TN with screw grips type 8253 (Fig. 3.5). The software TestXpert II registered the stress/strain data and curves. The tested specimens were placed in plastic boxes for optical analysis in a Leco Lx-31. The EBSD and EDS data acquisition was performed with FEI Teneo field emission scanning electron microscope (SEM) with Oxford EDS/EBSD capability and HKL Channel 5 software for pole figures. The PED-TEM data was obtained by preparing thin foils with a FEI Helios Focus Ion Beam (dual beam) system and a FEI Tecnai G2-F20 with a NanoMEGAS hardware and software package for the precession electron diffraction acquisition (PED) at a step size of 5 nm and angular resolution of 0.4°. Finally, the dislocation density calculation was performed by processing the orientation information from PED in a MATLAB algorithm [80].
Figure 3.4 Modified dog-bone shape specimens (a) WZ and (b) TMAZ.

Figure 3.5 ZwickLine Z2.5TN with screw grips type 8253
3.2 EBAM® Specimens Preparation and Ultrasonic Fatigue Test

The design of the ultrasonic fatigue specimen was performed using the commercial Finite Element Analysis (FEA) package COMSOL Multiphysics® 5.3 with the solid mechanics and electrostatic interfaces. The power supply, transducer, booster and horns are commercial products manufactured by Branson Ultrasonics. They were originally developed for ultrasonic welding applications, but the detailed technical information of those products was the input information in the FEA ultrasonic fatigue simulation. The converter (transducer) consists of 6 piezoceramic discs, stacked mechanically in series and electrically in parallel. Specimen geometry was drawn in Autodesk® Inventor Professional 2018 and imported to COMSOL, but the Catenoidal-608-001-021 (0.38” ½-20) horn, booster (Gold-101-149-057) and converter were drawn in COMSOL (Fig 3.6).

Figure 3.6 Final design of the specimen for ultrasonic fatigue test
The 6 piezoceramic discs in the converter are made of Lead Zirconate Titanate (PZT-5H) and the converter case, booster and horn are made of Ti-6Al-4V. The properties of these two materials including the elasticity, coupling and relative permittivity matrix of PZT-5H are those from the COMSOL material library. Internal damping was not applied for simplicity. The construction was fixed on the rear part of the converter and at the lower side of the specimen as illustrated in violet color in Figure 3.7. Two different studies were performed with the resonance system. First, a manual eigenfrequency search method was applied to find different eigenfrequencies around 20,000 Hz. Then, the right eigenmode (mode shape) of the system is selected among the different eigenfrequencies computed (Fig. 3.8). Finally, a frequency domain study was performed in a range of 200 Hz around the selected eigenfrequency to compute and verify the maximum stress and amplitude displacement of the cantilever. The shape and dimensions of the specimen were set in the eigenfrequency study by the trial-and-error method to produce the desired eigenmode (i.e. cantilever oscillation for bending test) and satisfying the shape and size requirements for the specimen to test the targeted microstructural feature on EBAM® ELI-Ti-6Al-4V. A mesh convergence study was performed using a sequence of element size of 25, 1, 0.5 and 0.05 microns.

Figure 3.7 The two fixed constraints of the resonance system
The mesh convergence and quality verification (i.e. element inversion) provides an optimized eigenfrequency value for the suitable eigenmode. The frequency domain study was performed to verify the maximum stress and amplitude of displacement of the cantilever at that eigenfrequency. The electric potential in the frequency domain study was set by trial and error method to predict and correlate the stress and displacement of the cantilever, but those results will be discussed in the corresponding section of Chapter 6. The eigenmode at Figure 3.8b shows a completely different behavior compared with figure 3.8a. The wrong eigenmode can produce a damage to any component of the resonance system, as in Figure 3.8a, the case of the actuator and the connection with the booster is undergoing the maximum oscillation.

Figure 3.8 (a) First eigenmode at 20,563 Hz and (b) second eigenmode at 20,283 Hz
Electron Beam Additive Manufacturing (EBAM®) blocks of ELI-Ti-6Al-4V (Extra Low Interstitials) were provided by Sciaky Inc. Based on COMSOL simulation, 20 specimens with 2 cantilever beams from each specimen were extracted via EDM. Figure 3.9 shows the extracted specimens with an average thickness of 1 mm and each cantilever was very carefully localized on the corresponding zones A and B of the microstructure.

![Figure 3.9 EDM extraction of specimens for the ultrasonic fatigue test](image)

The cantilevers in Figure 3.9 are both 1 mm thickness and according to the simulation, the thickness of the targeted cantilever should be half (0.5 mm). Any grinder in market as the GATAN Dimple Grinder do not produce flat surfaces. Therefore, a mini grinder/polisher was built (Fig. 3.10) to reduce those 500 mm (0.5 mm) and eliminate sharp edges or any residual brown tint of the EDM on cantilever gage sides. Fig. 3.11 shows a specimen with one cantilever of half thickness (500 μm) of the specimen. Optical microscope inspection of the specimens was performed with a Leco Lx-31. The power supply for the ultrasonic device is a Branson Model 2000xtt, 20 kHz, 1250 Watts, 110-120 V AC and 50/60 Hz. The actuator is a Branson 2000x ae, 2.0 inch cylinder, base mount and 40 inch column. The converter into the actuator
is a Model CJ20 with acorn connector. An aluminum purple booster of 20 kHz, 1:0.6 ratio (101-149-055) and a green booster of 20 kHz, 1:1 ratio (101-149-051). Two Catenoidal horns of 0.38” CAT ½-20 (608-001-021) and 0.5 CAT ½-20 (609-001-021).

Figure 3.10 Mini grinder/polisher to reduce thickness on cantilevers

Figure 3.11 Specimen for ultrasonic test with cantilever thickness reduction
3.3 EBAM® Specimens Preparation and Conventional Fatigue Test

Electron Beam Additive Manufacturing (EBAM®) blocks of ELI-Ti-6Al-4V (Extra Low Interstitials) and subsequently AM-β annealed were provided by Sciaky Inc. 20 rectangular beams of 40 mm length, 5 mm width and 4.5 mm thickness were extracted via EDM. Figure 3.12 shows the schematic locations of the extractions on the beams, following the nomenclature for A (10 specimens) and B (10 specimens) zones (microstructural features of EBAM® ELI-Ti-6Al-4V) illustrated in Figure 2.22. In this study, the purpose was to assess the microstructure influence on fatigue life, not the notch fatigue resistance. Therefore, the beams were polished by hand on 400-800 grit wet/dry SiC abrasive papers on all faces to remove the EDS recast layer. The edges were chamfered by manual polishing at approximately 45°. In addition, the top and bottom face of 5 mm width of each specimen was polished using a 0.04 colloidal silica suspension and one of these faces was etched with Kroll’s reagent to leave the testing areas exposed for further microscope analysis (Fig. 3.13).

Figure 3.12 EBAM® ELI-Ti-6Al-4V blocks with localization of zones A and B
Conventional fatigue tests were conducted by Westmoreland Mechanical Testing & Research, Inc. (WMT&R) in a four-point bend configuration in load control at room temperature on a servo-hydraulic machine employing a sinusoidal waveform operating at a test frequency of 60 Hz. The R-ratio was 0.1 and each specimen was set up so that the polished surface (mirror side) was in tension on the 23 mm support side and the etched surface was in compression on the 5.74 mm support side. Runout was defined as 1,000,000 cycles and any test reaching runout was discontinued. A single-arm extensometer was placed on the bend setup in order to help monitor for crack growth during the testing. An estimated crack initiation cycle count was determined by analyzing the position change of the single-arm extensometer as testing progressed for that specimen. The stresses were chosen by WMT&R, in order to develop the S-N curves for microstructure A and B.

Figure 3.13 Specimens ready for the conventional fatigue test
CHAPTER 4. INFLUENCE OF LFW Ti-6Al-4V MICROSTRUCTURE ON TENSILE MECHANICAL PROPERTIES

This chapter describes microstructural and tensile properties of parent material (PM), thermo-mechanically affected zone (TMAZ) and welded zone (WZ) that develop under linear friction welding (LFW) of Ti-6Al-4V. The typical microstructures of these three zones have been shown in Figures 2.16 and 2.17. In the first section of this chapter, microstructure and texture of the three distinct zones in LFW are described. Afterwards, the tensile test results on individual zones are compared to evaluate the microstructure and yield strength relationship. Precession electron diffraction (PED-TEM) scans are presented and discussed to assess the dislocation density contribution to the yield strength results. Finally, kernel average misorientation maps of all three LFW zones are shown and discussed.

4.1 Microstructure

Backscatter micrographs of the three distinct zones of LFW are shown in figure 4.1. The parent material (PM) has a bi-modal or duplex microstructure that consists of primary $\alpha_p$ grains surrounded by a lamellar microstructure of $\alpha$ laths in a $\beta$ matrix (Fig. 4.1a). The thermo-mechanically affected zone (TMAZ) is a distorted bi-modal microstructure as observed in Figure 4.1b.

![Figure 4.1 Microstructure zones across the LFW-Ti-6Al-4V joint](image)
The microstructure in the welded zone (WZ) has a random distribution of $\alpha$ laths in a $\beta$ matrix (Fig. 4.1c). As described in section 2.2, the welded zone reaches temperatures above the $\beta$-transus at phase II of LFW process. Therefore, this zone undergoes dynamic recrystallization that eliminates the bi-modal microstructure completely. This zone has two options of microstructure depending on the cooling rate. Coarse lamellar structure of parallel $\alpha$ laths delineated by the $\beta$ phase is obtained when the cooling rate is lower than 20°C/s, and random distribution of $\alpha$ and $\alpha'$ laths in a $\beta$ matrix is obtained when the cooling rate is between 20°C/s and 410°C/s [4, 17, 81]. The backscatter micrographs of WZ in Figure 4.1c and the in-plane view of the same zone in Figure 4.2 show an acicular morphology (i.e. needle like) and a very random lath orientation that corresponds to the $\alpha'$ martensitic microstructure. These results agree with those reported by several authors [18, 25, 28, 29] in linear friction welding.

Figure 4.2 Backscatter micrograph of in-plane ($YZ$) WZ of a dog-bone specimen

Martensitic $\alpha'$ laths are supersaturated in $\beta$-stabilizer element (i.e. Vanadium) due to the diffusionless transformation. Therefore, the vanadium content in the WZ $\alpha'$ laths should
be higher than the corresponding one at the $\alpha$ laths in the bi-modal microstructure on PM. Figure 4.3 shows five different vanadium composition measurement locations for $\alpha$ laths present in PM and WZ. The local composition was measured using standardless energy dispersive spectroscopy (EDS) and the values are reported in wt% in Table 4.1. The largest $\alpha'$ laths from WZ (~600 nm) were selected to avoid an interaction volume of the measurement larger than the lath itself. The higher vanadium content in the WZ laths also suggest the diffusionless or martensitic character of this transformation.

![Figure 4.3 Locations of vanadium composition measurement in (a) PM and (b) WZ](image)

<table>
<thead>
<tr>
<th>Measurement</th>
<th>WZ</th>
<th>PM</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>3.06</td>
<td>2.67</td>
</tr>
<tr>
<td>2</td>
<td>2.89</td>
<td>2.55</td>
</tr>
<tr>
<td>3</td>
<td>3.43</td>
<td>2.66</td>
</tr>
<tr>
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<td>3.56</td>
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<tr>
<td>5</td>
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<tr>
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<tr>
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<td>St. Deviation</td>
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</tr>
</tbody>
</table>
The bi-modal microstructure is present in the PM and TMAZ. However, the TMAZ exhibits a distortion or elongation of the primary $\alpha_p$ grains due to the nature of the LFW process. This variation in the primary $\alpha_p$ grains can be quantified using the software MIPAR™ by a mathematical concept called eccentricity. This concept describes how elongated or circular is a specific feature (i.e. equiaxed $\alpha_p$ grains). 0 is a perfect circle and 1 is a straight line [82]. Figure 4.4a shows an overview of PM and TMAZ regions. Inset (b) shows the severe deformation of the primary $\alpha_p$ grains by a higher bound of eccentricity value of 0.9678. On the other hand, inset (c) shows more round features with a lower bound of eccentricity value of 0.4873. This drastic change in shape of the microstructural features indicates that linear friction welding (LFW) is a process that involves severe plastic deformation.

Figure 4.4 Backscatter micrographs showing the overview (a) TMAZ (b) and PM (c)
4.2 Texture

To assess the influence of LFW on the microstructure development, EBSD mapping was performed on all three distinct zones. Figure 4.5 shows EBSD map and computed pole figures of PM. The (0001) pole figure suggest an alignment of basal poles with the x-direction (perpendicular to oscillation direction), but since it is the parent material, it is not texture as a consequence of LFW. The TMAZ orientation map is in Figure 4.6, and there is no evidence of texture at this zone. As mentioned before in section 2.2.2, Karadge et al. [25] reported random orientation in the TMAZ for lab scale blocks (26 mm x 13 mm x 70 mm) which is similar to our block size (31 mm x 31 mm x 59 mm). However, they reported texture at the TMAZ for large scale blocks with a reason still unknown.
The EBSD map and computed pole figures in Figure 4.7 also show a random orientation of the crystallites in the WZ. These results indicate that at least under the given processing parameters of these LFW-Ti-6Al-4V specimens, texture is not having an influence on tensile properties due to its random character on WZ and TMAZ. However, based on what was reported by Romero et al. and Karadge et al. [19, 25], the randomness in this WZ suggest a high axial pressure used in the manufacture of these LFW specimens.

Figure 4.6 IPF-Z EBSD map and computed pole figures of TMAZ
Figure 4.7 IPF-Z EBSD map and computed pole figures of WZ

The EBSD map in Figure 4.8 offers a large overview of the three zones with a width of 1 mm and a step size of 400 nm. It is noticeable that the indexing fraction decreases inasmuch the WZ is reached. This tendency indicates that the unindexing fraction is due and proportional to the degree of plastic deformation or lattice distortion from PM to WZ. The unindexed fraction is visualized in all EBSD maps as the dark areas. The orientation maps of PM, TMAZ and WZ from Figures 4.5-4.7 have a step size of 300 nm (according to feature size) and the unindexed fraction is 8.91%, 31.32% and 35.37% respectively. It is known that lattice distortion degrades the quality of the acquired diffraction pattern (i.e. Kikuchi bands)
and it is visible as a shift in some of the zone axis (i.e. Kikuchi band intersections) along with changes in the width of some Kikuchi bands (Fig. 4.9) [83]. For this reason, several Kikuchi diffraction patterns from each LFW zone were analyzed to observe the proportionality between unindexed fraction and plastic deformation or lattice distortion (i.e. proximity with the WZ). Figure 4.10 shows how the sharpness of the Kikuchi bands has a decreasing tendency from PM towards the WZ. In order to provide a more quantitative description of the Kikuchi bands quality for LFW zones comparison, a Rudin, Osher, and Fatemi (ROF) denoising was applied to the images to plot an intensity profile removing unwanted details, but preserving important details such as band edges.

Figure 4.8 IPF-Z EBSD map overview of the three LFW zones
Figure 4.9 An undistorted and distorted crystal with the resulting diffraction patterns [83]

Figure 4.10 Kikuchi pattern degradation along LFW zones
The intensity profile across the width of each image shows the variation of the pixel intensity or grey values as expressed in the plots of Figure 4.11. The PM and TMAZ share a common mean of 128 of pixel intensity, probably associated to the source of the diffraction patterns where the TMAZ is just a distorted PM microstructure. In contrast, the WZ has a different value of 126, probably associated to a completely different microstructure. However, the real difference in the sharpness of Kikuchi bands or image quality can be expressed as the standard deviation of the intensity profile. A high standard deviation (SD) of the intensity profile will indicate a high amount of variation or dispersion of pixel intensity. It means a more abrupt change at the edges of each Kikuchi band. Therefore, it represents a more defined diffraction pattern that has more opportunities to be indexed by the EBSD software. In Figure 4.11 the PM has a standard deviation of 9.78, the TMAZ has 7.25 and the WZ 6.79. This trend reflects the decreasing sharpness of Kikuchi bands or image quality from PM to TMAZ and WZ. This band sharpness correlation between single diffraction patterns of an EBSD map could suggest that a greater correlation between unindexed fraction from EBSD and lattice distortion represented by dislocation density could exist and be quantifiable.

4.3 Tensile Test Results

Four specimens per each individual LFW zone (i.e. WZ, TMAZ and PM) for a total of 12 out of 24 attempts were successfully tested. Figure 4.12 shows the stress/strain curve and the optical micrograph mosaic of PM1. The yield strength for that specimen is 955.3 MPa, and the ultimate strength is 1041.1 MPa at room temperature. These values are similar to those reported by Wanjara et al. [17], they also used the bi-modal microstructure as the PM where the yield strength is 980 MPa and the ultimate strength is 1030 MPa.
Figure 4.11 Intensity profile of Kikuchi patterns for all LFW zones
Considering the PM as a reference, now the WZ and TMAZ are analyzed to observe the comparative differences as consequence of LFW process. The stress/strain curve and optical micrograph mosaic of the WZ1 specimen after fracture are shown in Figure 4.13. At first glance, it is noticeable that there is an anomalous behavior of the curve after a strain of 0.28. It matches with the micrograph mosaic showing a change in the fracture pattern from the WZ to the TMAZ and PM. This is in agreement with literature and with the preliminary tensile tests in Section 3.1 and Figure 3.3, where PM always has a lower yield strength than WZ. This change in the fracture path modifies the real value of elongation and rupture strength. Therefore, and at least for WZ1 and WZ3 the elongation and rupture strength does not reflect the real properties of the WZ, however this deviation does not affect tensile properties such as yield strength and ultimate strength. On the other hand, WZ2 and WZ4 do not exhibit that anomalous behavior on the stress/strain curve and the micrograph mosaic does not show any fracture path deviation from the WZ as shown for WZ2 in Figure 4.14. The yield strength for the WZ1 is 1159.2 MPa and the ultimate strength is 1255.5 MPa, which is ~21% and ~20% greater than PM1. In addition, the corresponding values of WZ2 are ~16% and 18% greater than PM2.

Figure 4.12 Stress/strain curve and optical micrograph mosaic of PM1
On the other hand, the yield strength and ultimate strength of TMAZ1 are 1088.3 MPa and 1227.9 MPa respectively, which are about 14% and 18%, respectively, greater than PM1. These results indicate that the tensile properties are from high to low in the sequence of WZ > TMAZ > PM. However, the optical mosaic in Figure 4.15 for the TMAZ1 shows a similar phenomenon to the WZ1 in Figure 4.13. The fracture path on both sides starts at the TMAZ, but it moves downward toward the PM. Due to the small size of the TMAZ, it is very difficult to individually evaluate that zone. However, the values of yield strength and ultimate strength of the TMAZ1 specimen correspond to that zone. All other specimens, TMAZ2, TMAZ3 and TMAZ4 were given comparative values to WZ for the yield strength such as 1155.7 MPa for TMAZ2, 1149.1 MPa for TMAZ3 and 1169.9 MPa for TMAZ4. In addition, the optical mosaic of those specimens shows a clear deviation from the TMAZ as shown in Figure 4.16. Therefore, only TMAZ1 should be considered for the assessment of TMAZ tensile properties.

Table 4.2 shows the 12 specimens with their respective tensile properties. The average value of each LFW zone is highlighted in green except for the TMAZ (highlighted in red) where the valid tensile properties correspond just to TMAZ1 (also in green).
From Table 4.2 the first observation should be to compare the yield strength from the tensile test with the estimated by hardness test in Table 3.1. It is clear that the estimation from hardness test is overestimating the yield strength. For all the LFW zones the overestimation is 10~3%. However, when the LFW zones are compared between them in hardness test results and in tensile test results, we can see a close similarity.
Figure 4.16 TMAZ 2-3-4 Specimens showing the fracture path deviation from TMAZ

Table 4.2 Tensile properties of the three LFW zones

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Yield strength (MPa)</th>
<th>Ultimate strength (MPa)</th>
<th>% Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>WZ1</td>
<td>1159.25691</td>
<td>1255.56792</td>
<td>33.52</td>
</tr>
<tr>
<td>WZ2</td>
<td>1153.78784</td>
<td>1222.87109</td>
<td>27.96</td>
</tr>
<tr>
<td>WZ3</td>
<td>1215.81484</td>
<td>1305.81083</td>
<td>33.57</td>
</tr>
<tr>
<td>WZ4</td>
<td>1169.18978</td>
<td>1250.16400</td>
<td>24.71</td>
</tr>
<tr>
<td>Average</td>
<td>1174.51 ± 28</td>
<td>1258.60 ± 34</td>
<td>29.94 ± 4</td>
</tr>
<tr>
<td>TMAZ1</td>
<td>1088.31289</td>
<td>1227.86795</td>
<td>23.98</td>
</tr>
<tr>
<td>TMAZ2</td>
<td>1155.71436</td>
<td>1229.07235</td>
<td>40.65</td>
</tr>
<tr>
<td>TMAZ3</td>
<td>1149.11753</td>
<td>1245.47158</td>
<td>27.31</td>
</tr>
<tr>
<td>TMAZ4</td>
<td>1169.96669</td>
<td>1277.05675</td>
<td>31.75</td>
</tr>
<tr>
<td>Average</td>
<td>1140.78 ± 36</td>
<td>1244.87 ± 22</td>
<td>30.92 ± 7</td>
</tr>
<tr>
<td>PM1</td>
<td>955.35132</td>
<td>1041.10563</td>
<td>57.55</td>
</tr>
<tr>
<td>PM2</td>
<td>990.45837</td>
<td>1039.28580</td>
<td>58.37</td>
</tr>
<tr>
<td>PM3</td>
<td>969.67373</td>
<td>1023.50225</td>
<td>52.94</td>
</tr>
<tr>
<td>PM4</td>
<td>988.16144</td>
<td>1054.25114</td>
<td>56.85</td>
</tr>
<tr>
<td>Average</td>
<td>975.91 ± 16</td>
<td>1039.54 ± 12</td>
<td>56.43 ± 2</td>
</tr>
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</table>
The yield strength estimated by hardness on LFW zones is 1324 MPa for WZ, 1196 MPa for TMAZ and 1098 MPa for PM as shown in Table 3.1. These values indicate that WZ is about 20% greater than PM and TMAZ is about 9% greater than PM. On the other hand, from tensile test (Table 4.2) and taking the average values except for the TMAZ, we can see that the WZ is about 20% greater than PM and TMAZ is about 13% greater than PM. Therefore, even though the yield strength values calculated by hardness test are overestimated, the comparison among them has the same trend as the comparison among LFW zones in the tensile test. In other words, the yield strength in WZ is about 20% greater than PM for both cases and the TMAZ yield strength comparison with PM is underestimated by the hardness test just about 4% compared with tensile test.

The martensitic microstructure in the WZ is the primary reason for lower elongation (as shown in Table 4.2) and higher strength with respect to PM. Additive manufacturing techniques also produce martensitic microstructures due to the rapid solidification process and the reported yield strength values in literature are similar to those presented in this work. Vandenbroucke and Kruth [84] reported 1125 MPa and Vrancken et al. [85] reported 1110 MPa. However, Palanivel et al. [86] showed significant variations on the tensile properties on samples with complex geometry as-deposited by additive manufacturing (AM). They extracted dog-bone shape specimens from different regions of a Ti-6Al-4V impeller built by AM and reported yield strength values of 978 MPa, 1099 MPa and 1165 MPa. This variation in yield strength from AM specimens is a consequence of different cooling rate conditions, but those effects are not the purpose of this work. However, this suggest that the martensitic microstructure is not by itself the only one contributor to the higher yield strength and lower
elongation. Due to the nature of linear friction welding where severe deformation exists, it is possible that a high dislocation density is also playing a role in the tensile properties.

Dislocation density in polycrystalline materials can be calculated by several methods such as X-Ray diffraction (XRD) \[87, 88\], electron backscatter diffraction (EBSD) \[87, 89, 90\] and precession electron diffraction (PED) \[80\]. However, for materials with severe plastic deformation, the EBSD pattern quality degrades \[80, 91\]. X-ray diffraction can capture the bulk average dislocation density, but has problems at grain boundaries and triple junctions due to a lack of spatial resolution compared with PED \[80, 92\]. Therefore, PED is the selected option to calculate dislocation density because linear friction welding creates severe plastic deformation where dislocations accumulate near grain boundaries and triple junctions \[92\].

4.4 Dislocation Density

Lattice distortion is relieved by the formation of dislocations. In Figure 4.17 there are regions of dislocations with a net Burgers vector of zero (dislocations cancel each other due to opposite signs) and they are called statistically stored dislocations (SSDs). There are other regions of dislocations with a net Burgers vector of Non-zero that produces a lattice curvature or a local crystallographic orientation which are called geometrically necessary dislocations (GNDs). However, the resultant net Burgers vector of zero in SSDs is a consequence of the selected area which indicates that depending on the “window size” all dislocations can be GNDs \[83\].

Figure 4.18 shows the orientation maps, virtual bright field obtained by the TEM-PED system and the dislocation density maps with average values from the MATLAB algorithm. The first observation is the unexpected higher value of average dislocation density for PM of
$10^{17.0814} \text{ m}^2$ over both $10^{16.5405} \text{ m}^2$ for TMAZ and $10^{16.9142} \text{ m}^2$ for WZ. However, some considerations have to be analyzed to properly interpret these results.

Figure 4.17 Illustration of dislocations in subgrain regions adapted from [83]

Figure 4.18 TEM-PED orientation and dislocation density calculation results
In linear friction welding process the PM region does not undergo any plastic deformation. This means that there is not any generation of new dislocations as compared to TMAZ and WZ where plastic deformation does occur. A more detailed analysis of the bright field image of the PM and TMAZ reveals the presence of bending contours. These contours are localized, permanent bends in the specimen as a consequence of specimen (foil) preparation. They are commonly present in ductile thin foils and produce a lattice curvature (i.e. disorientation) that can lead to an overestimated dislocation density calculation [80, 93]. Figure 4.19 shows a better visualization of bending contours on PM. These results suggest that the values of dislocation density are not representing the pre-existence of GNDs.

![Figure 4.19 Bending contours in parent material region of LFW](image)

Despite a quantitative representation of the average dislocation density was not possible through TEM-PED and calculation, other indicators show the trend of the degree of deformation for the three LFW regions in the order of PM < TMAZ < WZ. The unindexed fraction mentioned in Section 4.2 and Figure 4.8 shows that trend and HKL Channel 5 software can plot kernel average misorientation maps (KAM) using the EBSD orientation information of each zone. KAM show local misorientation associated to plastic deformation by averaging
crystal orientation differences between a host pixel and its adjacent pixels [94]. Figure 4.20 shows the KAM maps for the three LFW regions. The scale color (blue to red) represents the degree of misorientation associated to plastic deformation from a 1st order neighbor (3 x 3 pixel) with a threshold angle of 5°. The trend is again clear on the same direction of unindexed fraction of EBSD data (PM < TMAZ < WZ). Possibly a quantitative description of this trend can be achieved by a bulk dislocation density measurement via X-Ray diffraction with the challenge of small size availability of the LFW regions, specially the TMAZ with a width of just ~200 μm.

Figure 4.20 KAM maps of the three linear friction welding zones
4.5 Conclusions

The tensile test results show the yield strength of WZ is about 20% greater than PM and TMAZ is about 13% greater than PM. Even when evidence of dislocation accumulation as a consequence of deformation was not possible via TEM-PED and calculations, the qualitative description given by the unindexing fraction of EBSD and the KAM maps suggest evidence of strain hardening. The strain hardening inherent to the LFW process together with the martensitic microstructure constitute the explanation for the tensile test results. The PED approach to measure the dislocation density offers the high spatial resolution required to capture those dislocations generated by highly deformed or UFG (ultrafine grained) materials. However, this approach works under the assumption that bend contours are unlikely in highly deformed high-strength materials and among the LFW zones only the WZ would qualify. Therefore, it is proposed that in the future a bulk dislocation density calculation should be performed via X-ray diffraction or another alternative method.
CHAPTER 5. ULTRASONIC FATIGUE TESTING OF EBAM®-Ti-6Al-4V

This chapter describes the results for the ultrasonic fatigue test on Ti-6Al-4V specimens from the modeling perspective using the finite element analysis software COMSOL multiphysics®. Then, it extends the prediction and relationship between stress and displacement through an analytical solution using the strain energy method. Finally, it describes the preliminary experimental results on the Branson ultrasonic equipment and explains the differences and similarities with the theoretical results.

5.1 Modeling of the Ultrasonic Fatigue Test

The ultrasonic test setup was modeled using the finite element software COMSOL Multiphysics® as described in Section 3.2. The calculated eigenfrequency for the desired mode shape oscillation was 20,011 Hz as shown in Figure 5.1. In the frequency domain study a frequency range around eigenfrequency was set (19,911 Hz - 20,011 Hz – 20,111 Hz, meaning the eigenfrequency value ±100 Hz). The maximum stress and displacement amplitudes are at the eigenfrequency, 20,011 Hz, and the electric potential was set by trial and error method to produce the stress amplitude values observed in literature for a fatigue test (i.e. S-N curve) in a Ti-6Al-4V alloy [37, 49].
Figure 5.1 Eigenfrequency of the resonant system for the desired mode shape oscillation

The stress and number of cycles curve (S-N curve) has a maximum and minimum stress amplitude value to fail at different number of cycles. Based on Ti-6Al-4V properties from the COMSOL data bank, the model predicts 486.2 MPa with a cantilever deflection of 20.6 µm as the stress lower bound (Fig. 5.2). The upper bound shows a stress amplitude of 972.4 MPa with a cantilever deflection of 41.2 µm (Fig. 5.3).

Figure 5.2 Stress lower bound showing the stress profile on cantilever surface
Figure 5.3 Stress upper bound showing the stress profile on cantilever surface

Figures 5.1 and 5.4 shows that the oscillation of the cantilever is not completely straight. It is clear that the bottom part of the square section moves first and has a wider oscillation compared with the top square part. This observation is important because it will determine the experimental method for measuring the deflection on the cantilever, which is the starting point to obtain the experimental stress. Eddy current or capacitive proximity sensors
used by Morrissey et al. [38] in ultrasonic tension/compression fatigue test have an electromagnetic field that changes with the variation of distance from a metallic target (cantilever). This change generates a voltage that is converted into a displacement measurement. These sensors would be able to measure the displacement and the number of oscillations accurately at 20 kHz frequency. However, the target should be three times larger than the coil diameter of the eddy current sensor or 30% larger that the capacitive sensor. This limitation narrows the deflection measurement on the 5 mm square section of the cantilever, which is a good approximation, but not the most accurate deflection at the narrow section of the cantilever (our target) (Fig 5.4). Therefore, a new method for the deflection measurement should be defined in the future. Table 5.1 shows the data from eleven simulations with different electric voltage (V) to obtain the relationship between stress amplitude and displacement from the finite element analysis.

Figure 5.4 Cantilever deflection in the lower (486.2 MPa) (a) and upper (972.4 MPa) (b) bounds

In addition to the numerical solution (i.e. finite element analysis) to obtain stress/displacement relationship, an analytical solution can also provide that relationship for
further comparison with the experimental results. The strain energy method is a standard method to estimate deflection of constant or varying cross section beams. The work done to produce deflection is equated to the strain energy stored by the beam [95]. This method includes area moment of inertia of all involved sections. However, due to the non-straight deflection of the cantilever as mentioned before, the analytical calculation will be based just in the 3 mm length beam with a square cross section of 500 μm.

Table 5.1 Stress/displacement relationship from COMSOL Multiphysics®

<table>
<thead>
<tr>
<th>Displacement (μm)</th>
<th>Stress (MPa)</th>
<th>Comsol Voltage (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20.61</td>
<td>486.18</td>
<td>25.0</td>
</tr>
<tr>
<td>22.67</td>
<td>534.8</td>
<td>27.5</td>
</tr>
<tr>
<td>24.73</td>
<td>583.41</td>
<td>30.0</td>
</tr>
<tr>
<td>26.79</td>
<td>632.03</td>
<td>32.5</td>
</tr>
<tr>
<td>28.85</td>
<td>680.65</td>
<td>35.0</td>
</tr>
<tr>
<td>30.92</td>
<td>729.27</td>
<td>37.5</td>
</tr>
<tr>
<td>32.98</td>
<td>777.88</td>
<td>40.0</td>
</tr>
<tr>
<td>35.04</td>
<td>826.5</td>
<td>42.5</td>
</tr>
<tr>
<td>37.1</td>
<td>875.12</td>
<td>45.0</td>
</tr>
<tr>
<td>39.16</td>
<td>923.73</td>
<td>47.5</td>
</tr>
<tr>
<td>41.22</td>
<td>972.36</td>
<td>50.0</td>
</tr>
</tbody>
</table>

This method is assuming a point load on the free end of the cantilever section (i.e. 3 mm length section) and that the bending moment is transferred to the fixed base [95]. We know that this is not the specific case for the cantilever oscillation, but it offers a different point of view for the stress at the base of the cantilever and the deflection of the entire cantilever. Equation 5.1 shows the work done to produce deflection and equation 5.2 shows the strain energy stored by the cantilever. Equating 5-1 and 5-2, substituting values, replacing W (point
load) in function of \( \sigma \) (stress) using 5-3 and solving the equation we obtain the values of stress and displacement in table 5.2.

\[
\text{work done} = \frac{1}{2} (W) (\delta) \quad \text{Eq. 5-1}
\]

\[
E = \int_0^3 \left( \frac{(Wx)^2}{2EI} \right) dx \quad \text{Eq. 5-2}
\]

\[
\sigma = \frac{WD(h/2)}{l} \quad \text{Eq. 5-3}
\]

\( W \) = point load

\( \delta \) = total deflection at free end

\( E \) = Young’s modulus

\( I \) = area moment of inertia

\( D \) = length of cantilever

\( h \) = thickness of the cantilever

Table 5.2 Stress/displacement relationship from strain energy method

<table>
<thead>
<tr>
<th>Displacement (µm)</th>
<th>Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>51.63</td>
<td>486.18</td>
</tr>
<tr>
<td>56.79</td>
<td>534.80</td>
</tr>
<tr>
<td>61.95</td>
<td>583.41</td>
</tr>
<tr>
<td>67.12</td>
<td>632.03</td>
</tr>
<tr>
<td>72.28</td>
<td>680.65</td>
</tr>
<tr>
<td>77.44</td>
<td>729.27</td>
</tr>
<tr>
<td>82.61</td>
<td>777.88</td>
</tr>
<tr>
<td>87.77</td>
<td>826.5</td>
</tr>
<tr>
<td>92.93</td>
<td>875.12</td>
</tr>
<tr>
<td>98.10</td>
<td>923.73</td>
</tr>
<tr>
<td>103.26</td>
<td>972.36</td>
</tr>
</tbody>
</table>
This analytical approach is showing also a linear relationship, but the corresponding displacement is about double compared to the numerical solution. The numerical and analytical approaches have different assumptions and physics involved, which could explain the differences in the results. However, these results are a theoretical prediction of stress and deflection that can be expected and compared to the experimental ultrasonic fatigue test. Figure 5.5 shows the stress vs displacement curve for both scenarios.

Figure 5.5 Stress/ displacement of cantilevers from numerical and analytical method

5.2 Preliminary Experimental Ultrasonic Fatigue Test

Based on modeling and theoretical results in the previous section, preliminary test on cantilevers is performed. The first preliminary test has a cycle time of 6.814 s which corresponds to 5 s for the contact with the specimen (weld time using the terms of the equipment that is originally an ultrasonic welding machine) and 1.814 s for the movement of the stack. Due to availability at Branson Ultrasonics facility, the booster used for the test was
a green 1:1, EDP No. 101-149-051 and the horn was a Catenoidal-609-001-021 (0.5” ½-20). The peak power (Pkpwr) is 10.9% of the potential power of the power supply during the cycle. The power supply is rated at 4,000W, so it was 436W. The peak-to-peak movement at the horn face (SetAmpA), always expressed as a percentage of the maximum, is 75%. The resonance frequency of the system is 19,792 Hz. However, this first preliminary test broke the specimen into pieces within the first 0.25s of the contact time. Figure 5.6 shows the specimen with cracks in all corners.

Figure 5.6 First preliminary ultrasonic fatigue test on cantilever

The complete failure of the first preliminary test provides information about time and power for the second test. Figure 5.7 shows the cantilever with the reduced 500 μm thickness oscillating (a) and then within 0.25s flying apart (b) after fracture. This second preliminary test has a cycle time of 0.25s, same booster and horn as for the first test, but different conditions of power and amplitude. The Pkpwr is 2.3% of the power supply during the cycle, the test amplitude (SetAmpA) is 50% and the resonance frequency is 19,800 Hz. Following the second
preliminary fatigue test on the reduced 500 μm thickness cantilever (microstructure B), another attempt is performed in order to observe the behavior of the other cantilever on the same specimen that has a thickness of 1 mm. From the COMSOL model it is not expected to oscillate in the right mode shape due to the different thickness. As shown in figure 5.8, the cantilever undergoes torsion and oscillations that are different to the designed mode shape. However, this extra test provides information to assess the proximity of modeled to experimental results.

Figure 5.7 Second preliminary ultrasonic fatigue test in the 500 μm thickness cantilever (a) before and (b) after failure

Figure 5.8 Second preliminary ultrasonic fatigue test in the 1 mm thickness cantilever (a) before and (b) after failure
5.3 Conclusions

The first results to analyze are the eigenfrequency and mode shape predicted by the finite element method compared with the experimental. In the experimental ultrasonic fatigue test a different booster (i.e. green instead of gold) and horn (Catenoidal of 0.5” instead of 0.38”) was used due to availability at Branson Ultrasonic facilities. However, the mode shape in the second preliminary test as shown in figure 5.7a is as expected. In other words, the cantilever is bending up and down (R = -1) as needed to perform the fatigue test. On the other hand, the COMSOL modeled eigenfrequency is 20,011 Hz and the experimental is 19,800 Hz, a difference of ~1% (211 Hz). It is known that differences of ~1% could lead to a different mode shape of oscillation as shown in Figure 3.8, where a difference of 280 Hz in the calculated eigenfrequency shows a mode shape affecting more the actuator and connection with booster than the specimen. However, considering the differences in booster and horn for the modeled and experimental test, the prediction of specimen shape and dimensions are close enough to provide the right mode shape of oscillation.

The stress and deflection are important predictions from the model since the deflection can be used to estimate the targeted stress on cantilever to construct the S-N curve. The theoretical deflection for a targeted stress on cantilever provides a guidance for the amplitude required at the horn face. This amplitude is determined by the input power (power supply), the gain of booster and horn and the test amplitude (SetAmpA) selected in the front panel control of the equipment. Therefore, it will be very important to compare an experimental stress/deflection relationship with the predicted by COMSOL Multiphysics® and the strain energy method.
CHAPTER 6. CONVENTIONAL FATIGUE TESTING ON EBAM®-Ti-6Al-4V

This chapter describes the four-point bending fatigue test results on electron beam additive manufactured Ti-6Al-4V specimens with specific features evaluation. Crack initiation and propagation analysis is discussed based on optical and electron micrographs and comparison of microstructures A and B is performed. Finally, conclusions about the comparison of the two microstructures and stress life data points are described.

6.1 Conventional Fatigue Test Results on EBAM®-Ti-6Al-4V

The four-point bending fatigue test was conducted by Westmoreland Mechanical Testing & Research, Inc. at room temperature on a servo-hydraulic machine employing a sinusoidal waveform as described in Section 3.3. Figure 6.1 shows the captured features for specimens tested to evaluate the selected A and B microstructures. In Figure 6.2, the stress life data curves reported by Westmoreland, Inc. shows that microstructure A (blue) has a better fatigue performance than B (red). The analysis to explain how the microstructure affects that behavior can be divided into two perspectives, crack initiation and crack propagation.

![Figure 6.1 Microstructures A and B from EBAM®-Ti-6Al-4V](image-url)
6.2 Crack Initiation Perspective

Cyclic loading of metals undergoes plastic strain localization which leads to the formation of bands or concentrated slips. It was established that locations of bands remained visible after electro-polishing and the slip activity re-occurred at the same locations if the fatigue test continued [96]. This means they are structurally different from the surrounding material and that is why they are called persistent [97]. There are three basic types of crack initiation; these are initiation at fatigue slip bands, initiation at grain and twin boundaries and initiation at inclusions, pores and other inhomogeneities. Initiation at fatigue slip bands is a localization of cyclic slips that results in a creation of a surface with a hill-and-valley
topography [96, 98]. Initiation at grain and twin boundaries is the interaction of large-angle grain boundaries at sites where the slip bands (intrusions) impinge on them. Finally inclusions, pores and other inhomogeneities can also interact with the intrusions of the well-developed persistent slip bands (PSBs) to initiate a crack. Figure 6.3 shows a schematic representation of the different stages of surface relief evolution to form persistent slip bands (PSBs). One example of crack initiation is presented in Figure 6.4 of specimen B-2 (tested at 700 MPa). Those secondary images show the intrusion, crack propagation and different size of slip bands near the crack.

Figure 6.3 Stages of surface relief evolution to form (b) ribbon-like extrusions and (d) mature PSBs with well-developed intrusions at both PSB-matrix interfaces.
Figure 6.4 Secondary electron micrographs of one side of specimen B-2 showing a crack initiation site

6.2.1 Comparing microstructure A with B under similar conditions

In order to compare the A and B microstructures, it is necessary to select specimens under similar conditions of stress amplitude. B-9 and A-18 were tested at 800 MPa, but B-9 failed at 44,884 cycles while A-18 failed at 220,108 cycles. Slip band length quantification was performed using the software MIPAR™. Figure 6.5 shows one example of the process on MIPAR™ to measure the slip length on one specific region on samples B-9 and A-18. A manual elimination of some features was necessary to calculate the slip length on different images because they did not correspond to slip bands but were similar to them in image processing. Figure 6.6 shows the comparison between B-9 (5487 µm) and A-18 (6468 µm) of slip length, meaning A-18 has slip bands about 15% greater than B-9. However, the stage of formation of the slip bands as explained in Figure 6.3 is more advanced for B-9 compared to A-18. The different orientations and sizes of slip bands on Figure 6.4 and the different degree of slip formation (i.e. surface relief evolution) of B-9 compared to A-18 in Figure 6.6, suggest
that there is a preferential $\alpha$ colony orientation respect to the loading direction for the slip band formation. A closer backscatter image in figure 6.7 shows that difference in stages of slip band formation for both specimens.

![Figure 6.5 One example of the slip length measurement on MIPAR™](image)

![Figure 6.6 Secondary-electron images to compare B-9 and A-18](image)
Figure 6.7 Backscatter images of B-9 and A-18 showing the different stages of slip band formation

More observations on specimen A-18 at the etched surface, reveal more visible slip bands. Near the fracture surface, some \( \alpha \) colonies were presenting visible slip bands formation, but some others did not. Figure 6.8a shows one \( \alpha \) colony with the laths oriented perpendicular to the fracture surface with a high concentration of slip bands. On the other side, adjacent \( \alpha \) colonies do not show visible slip band formation. A closer view in figure 6.8b reveals the existence of dark and small early stage slip bands on the \( \alpha \) colony next to the one highly populated with shiny slip bands. Figure 6.8b also shows small white features inside the \( \alpha \) laths, which requires more investigation but is not the focus of this thesis.

Figure 6.8 Secondary images of A-18 (etched side)
6.3 Crack Propagation Perspective

These EBAM®-Ti-6Al-4V specimens have fully lamellar microstructure where slip or crack propagation barriers are $\alpha/\beta$ interfaces $< \alpha$ colony boundaries $< \text{prior-beta grain boundaries}$, in that order of strength. As mentioned in Section 2.1.4.2, $\alpha/\beta$ interfaces can transfer the slip by the parallel slip system $(110) \{1\bar{1}1\} \beta (0002) \{11\bar{2}0\} \alpha$ or others that are off by only $\sim 10^\circ$ or less. Figure 6.9a shows an example of $\alpha/\beta$ interface acting as a barrier in specimen A-1, with the crack having to propagate following this interface as it cannot traverse it. $\alpha$ colony boundaries in Figure 6.9b are boundaries of different orientation clusters of $\alpha$ laths that represent a stronger obstacle for slip or crack propagation compared to $\alpha/\beta$ interfaces. Prior-beta grain boundary in Figure 6.9c is the strongest barrier for slip or crack propagation due to the presence of continuous $\alpha$ layers and the orientation transition to a completely different oriented $\beta$ grain with different internal variants of $\alpha$ colonies.

6.3.1 Comparing microstructure A with B under similar conditions

In order to compare the microstructure A with B under the crack propagation perspective, it is necessary, once again, to select specimens under same conditions of stress amplitude. B-2 and A-12 were tested at a maximum stress of 700 MPa, but B-2 failed at 101,753 cycles and A-12 reached the limit of 1,000,000 cycles (runout). Optical images of the specimen A-12 revealed the presence of prior-beta grain boundaries oriented perpendicular to the fracture direction. In addition to this orientation of those grain boundaries in A-12 unfavorable for crack propagation, they are statistically more present in specimen A-12 compared to B-2. Figure 6.10 shows those optical images and comparison. Additionally, optical images in Figure 6.11 comparing B-9 with A-18 also show the same trend as described for the previous set of samples for crack propagation.
Figure 6.9 Examples of slip or crack propagation barriers on fatigue specimens (a) A-1 (b) B-2 and (c) B-9
Figure 6.10 Optical images of bending specimens comparing B-2 with A-12

Figure 6.11 Optical images of bending specimens comparing B-9 with A-18
6.4 Conclusions

The stress life data indicates in general terms the better fatigue performance of microstructure A compared to microstructure B. Optical and electron microscope analysis suggests two perspectives to explain such behavior. From crack initiation perspective, the slip band formation is in a more advanced stage in microstructure B compared to A due to the more random $\alpha$ colony orientation that provides a better opportunity to hcp slip systems being aligned with the loading direction in the bending test. From crack propagation perspective, the orientation of the columnar grains provide a statistically more presence of prior-beta grain boundaries perpendicular to the slip band lines or crack propagation. These two factors may explain the better performance of microstructure A.

Excluding those 5 specimens (3 of microstructure B and 2 of microstructure A) who never failed (runout) from the stress life data curves (Fig.6.2), we can observe that all B samples failed at lower maximum stress than A samples except B-9 (800 MPa) and B-8 (775 MPa). However, the A specimens with the same maximum stress of those two B exceptions such as A-18 (800 MPa) and A-14 (780 MPa) failed at a much higher number of cycles than the similar samples of microstructure B. Another important observation is that above 700 MPa there is not a single runout specimen. The two specimens of microstructure B that failed below 700 MPa (B-2 and B3) may contain random microcracks or defects that are inherent to the manufacturing process.
CHAPTER 7. CONCLUSIONS AND SUGGESTIONS FOR FUTURE RESEARCH

In this work, the microstructure development as a consequence of linear friction welding process on Ti-6Al-4V specimens is evaluated. Most research on this process has been done with failure at the parent material as the weakest region, and to the author’s knowledge this is the first time that microstructure evaluation and tensile tests focus on individual zones of the LFW process has been investigated. The tensile tests revealed that the yield strength of WZ is ~20% and TMAZ ~13% greater than PM. Bending contours and not enough plastic deformation in PM and TMAZ did not allow a successful TEM-PED and calculation approach to assess the dislocation density in the three LFW zones. However, qualitative approaches such as unindexing fraction from EBSD maps and kernel average misorientation (KAM) maps, suggest the higher degree of plastic deformation of WZ > TMAZ > PM. Therefore, strain hardening and the martensitic microstructure constitute the explanation for the tensile test results. It is suggested that in the future, average dislocation density should be performed via X-Ray diffraction or alternative methods.

Ultrasonic bending fatigue test adaptation on a commercial welding equipment was designed to reach very high cycles regime and reduce the sample size to assess localized features on electron beam additive manufactured Ti-6Al-4V specimens. The available research on this area has been focused on tension/compression, rotational stress, but not bending application to assess general microstructures and no localized features. Preliminary ultrasonic fatigue tests confirmed the resonance frequency, shape and dimensions of specimens predicted from the finite element analysis on COMSOL Multiphysics®. Additional experimentation to obtain stress and deflection data will be very important to compare with the predicted and with the behavior of conventional fatigue test in Chapter 6.
Conventional fatigue tests on electron beam additive manufactured Ti-6Al-4V specimens were performed to evaluate individual features in the microstructure and to establish a blank of comparison with a future ultrasonic fatigue test on the same type of material. The further validation of the ultrasonic fatigue test with the conventional will lead to the fatigue evaluation of the individual zones in linear friction welding (LFW) of Chapter 4. The previous research about EBAM®-Ti-6Al-4V material has been focused on tensile properties, but no research work has been reported on evaluating localized fatigue properties. The microstructure A showed a superior fatigue behavior than B based on both crack initiation and crack propagation perspectives. It is suggested to change the orientation of specimen’s extraction to see the slip lines and crack propagation behavior. In addition, a more detailed analysis of extrusion/intrusion phenomena on different specimens should be performed.
CHAPTER 8. REFERENCES


