3D Characterization of Ti-6Al-4V using the TriBeam System

Master Thesis

Marcus Johnson*

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Supervisors:
John Ågren* and Tresa M. Pollock

Assistant supervisors:
Peter Kolmskog* and McLean P. Echlin

* Department of Materials Science and Engineering, Royal Institute of Technology, KTH
Brinellvägen 23, 100 44, Sweden

Materials Department, University of California, Santa Barbara, UCSB
CA 93106-5050
Abstract

Three-dimensional characterization of fully equiaxed Ti-6Al-4V has been performed using the recently developed TriBeam system. To date, no general model for full prediction of Ti-6Al-4V tensile properties as a function of empirical microstructural parameters exist. To develop such a model would require knowledge about the relevant deformation mechanisms and also detailed information about the microstructure of the material. As a thrust to develop such a model a large three-dimensional dataset was collected, reconstructed and analyzed, in order to extract statistical information about microstructural parameters. It was found that the material contained heavily textured regions with α grain boundaries that have misorientation of 1° to 2° or less. The low grain misorientation angle required segmentation parameter optimization, which included the integration of a grain size filter and a thorough exploration of the segmentation parameter space. The α grains are shown to be volumetrically smaller in textured regions compared to bulk, while β grains showed an even grain volume distribution throughout the structure. The TriBeam system collected the dataset faster than any other available serial sectioning methods and is described in detail with its subsystems and operating conditions. The extracted statistical information is presented in detail numerically and visually in this report.
Acknowledgements

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1 Motivation

Titanium alloy Ti-6Al-4V is a widely used alloy in several types of high performance applications such as in aerospace, turbine engines and marine applications. Even though the alloy is so prevalent the relationships between microstructure and mechanical properties are still not fully understood. Reliable predictions of titanium's mechanical behavior, based on microstructural observations, are difficult because of the complicated microstructure and the highly textured nature of the material. A structure property model would need to take specific microstructural parameters into consideration and mathematically link them to the desired mechanical properties. Such a model would be useful to better understand the connections between the processing route of titanium and its final properties. Research has shown that there are correlations between measured microstructural parameters and mechanical properties such as tensile strength (1). In order to develop an explicit structure property model, first an understanding of the independent microstructural parameters it should contain is required. As a thrust to better understand these parameters, extraction of microstructural parameters from Ti-6Al-4V has been performed.

Measurements of microstructural parameters are conventionally made by two-dimensional observations of mechanical sectioned samples. It is known that two-dimensional observations can only provide limited spatial information (2) (3). Alternately tomography can be used to obtain improved information about microstructure. The TriBeam is a recently developed technique to perform serial sectioning of materials (4). It uses a femtosecond laser to iteratively ablate layers from a material surface, with intermediate imaging steps. Imaging modes include an electron backscatter diffraction (EBSD) detector, energy dispersive X-ray spectroscopy (EDS), backscatter electron (BSE), secondary electron (SE), and ion induced secondary electron (ISE). In this research the Tribeam system was used to obtain crystallographic information from titanium samples using backscattered electron diffraction imaging. The two-dimensional EBSD slices were then reconstructed into three-dimensional texture models.
The aim of this work is to:

- Produce three-dimensional datasets for alloy Ti-6Al-4V and describe the TriBeam operating conditions to do so.
- Analyze dataset and gather microstructural statistics
- Through the performed work obtain higher skills within the field of advanced microscopy and titanium analysis

This project was performed as a final degree project at the Department of Materials, Royal Institute of Technology, Stockholm. All work was performed during September 2012 to January 2013, at the Materials Department, University of California, Santa Barbara.

2 Background

In the following sections, Ti-6Al-4V will be discussed as well as a review of the tomographic method used to analyze this material and the protocols for reconstruction and analysis of three-dimensional datasets.

2.1 Titanium

Even though titanium is the fourth most abundant structural metal in the earth’s crust, it is never found in its pure state (1). It is usually found in low concentrations in the minerals ilmenite (FeTiO₃) and rutile (TiO₂), and has to be extracted and further processed.

Titanium and its alloys are expensive due to the cost of the processing steps required for refinement of ore to pure metal. The main process for producing titanium is the Kroll process, which contains several different energy-intensive process steps (5). This is a batch type of process with numerous high temperature steps. Research is being done to find methods to produce titanium in a continuous process, which could decrease the energy needed for production and lower the market price (6).

Due to the high cost of titanium it is primarily used in products where the mechanical properties and weight are of great importance. Therefore titanium has mostly been used in industries that require the best the market can offer regardless of price, such as aerospace and chemical industries (1). Titanium is the heaviest of the light metals with a
density of 4.51g/cm³ and its high specific strength and excellent corrosion resistance makes it a given choice for many applications. Figure 1 shows strength to density ratio for titanium alloys compared to other materials.

Figure 1 Diagram showing strength - density for different materials (7).

2.1.1 Microstructure
Pure titanium has two types of microstructure, hexagonal close packed (HCP) and body-centered cubic (BCC), making it an allotropic metal. These crystalline structures are illustrated below in Figure 2.

Figure 2 Illustration of titanium structures and close packed planes within them, HCP β-phase to the left and BCC α-phase to the right. (1)
The β-transus temperature for pure titanium occurs at 882±2°C (1) and that is the temperature at which the microstructure will change between these two structures. The HCP, α phase is stable below the transus temperature, and above the transus temperature the BCC, β phase is the stable phase. Figure 3 shows the Ti-Al phase diagram, where the two phases are seen separated for low Al-content.

![Figure 3 Ti-Al binary phase diagram showing the α-β transus for low aluminum content (8)](image)

The most closely packed slip plane in BCC is the (110), which during β→α transformation becomes the basal plane (0001) in HCP. There are 12 slip systems in BCC and therefore are equally many directions for the HCP to grow in (1), during phase transformation. This makes the 2D pattern similar to a basket weave and is sometimes called basket-weave structure (1), this can be seen further down in Figure 5f. Depending on application, different alloys are used with different microstructure and properties, which can be tailored alloying element and thermo-mechanical treatment. The ability to control phase fraction and grain properties is the key to determining what mechanical properties can be obtained (9).

### 2.1.2 Alloy Ti-6Al-4V

The alloy investigated in this project is Ti-6Al-4V. It is the most widely used Ti-alloy and it was originally developed in 1954 (8). The two main reasons for its widespread use is its good balance of properties. It is by far the most tested and intensively developed titanium alloy (1). Depending on what phase is dominant in the post-heat treated alloy, titanium alloys are mainly classified as *α*-alloys, *α+β*-alloys and *β*-alloys (1). Alloy Ti-6Al-
4V is classified as an α+β-alloy. The aluminum is a strong α-stabilizer, increasing the transus temperature to 995°C (8), while vanadium is a β-stabilizer (1). A pseudo-binary phase diagram of alloy Ti-6Al-V is shown below in Figure 4. A pseudo-binary phase diagram shows a ternary system with the ratio of two constituents fixed, projected as a binary phase diagram.

![Figure 4 Pseudo-binary phase diagram of Ti-6Al vs V. MS is martensitic start temperature (1)](image)

Alloy Ti-6Al-4V is used in a variety of applications (e.g. deep sea drilling risers, golf club driver heads, fan/compressor blades and airframes) due to its high strength (900-1200 MPa) and high fatigue resistance. The range in strength for the alloy depends on many different parameters such as processing route, heat treatment, aging and which direction the material is tested in. Figure 5 shows several types of microstructures that can exist in Ti-6Al-4V.
Figure 5 Different types of microstructure that can occur in Ti-6Al-4V, depending on thermomechanical treatment. White phase is α-phase and darker phase is β-phase (9).
The microstructure of $\alpha+\beta$-alloys can be classified into three major types; fully lamellar, fully equiaxed and duplex (bimodal), see Figure 6.

![Image](image_url)

**Figure 6** (a) Optical micrograph of fully lamellar microstructure in Ti-6Al-4V. (b) Backscatter electron image of fully equiaxed microstructure in Ti-6Al-4V. (c) Backscatter electron image of bimodal (duplex) microstructure in Ti-6Al-4V (10)

The parameter that most strongly controls final microstructure is the cooling rate from $\beta$ phase to $\alpha$ phase region in the phase diagram (10).

When slow cooling through the transus temperature, the $\alpha$ phase will nucleate and precipitate initially at $\beta$ phase grain boundaries and exist as $\alpha$-layers along the boundaries, and then later grow into the prior $\beta$ grain as parallel plates (8). Each parallel
plate with same orientation is considered an α colony. The colonies in a β grain will grow until they meet colonies from other grain boundaries. Between the plates there will be retained β phase, resulting in a fully lamellar structure. The fully lamellar structure can vary from a coarser colony type to a finer martensitic type, depending on slow or fast cooling rate respectively. The defining features between the two types are size of α-lamellae, size of α colonies and thickness of α phase at the prior β grain boundaries (10), example of fully lamellar microstructure can be seen in Figure 5f and Figure 6a.

High temperature deformation of fully lamellar microstructure performed below the β-transus temperature will break down the α plates into small segments. Subsequent heat treatment in the α+β-region performed after hot working will recrystallize the α phase segments and result in an equiaxed spherodized structure (10). Slow cooling from this state, such as furnace cooling, down to room temperature will allow growth of α phase until only small amounts of β phase remains as separating phase around the α phase. While faster cooling such as air-cooling will not allow the same α phase growth, instead transformation of β phase into short α-lamellae surrounded by retained β phase will occur (10). This microstructure of equiaxed α grains and α-lamellae surrounded by β-matrix is called bimodal or duplex. Compared to fully lamellar structure, bimodal structure shows increment in yield strength, ductility, fatigue-crack-initiation resistance and also slower fatigue-crack-propagation rate. Making it a suitable microstructure in jet engine applications such as turbine discs, fan and compressor blades (8).

2.1.3 Mill-annealing
The alloy Ti-6Al-4V used in this project was received in mill-annealed condition. It is a common general-purpose treatment that covers a range of processing conditions. The microstructure from this treatment can vary between manufacturers or even between batches (8). Figure 7 shows the processing route for mill-annealing. The mill-annealed microstructure has been described as containing incompletely recrystallized α-phase with low volume fraction of small β-phase (9). The cooling rate of the homogenization step affects the α-grain size. During deformation parameters such as; number of heats, heating time, grade of deformation and cooling rate affect the degree of recrystallization and therefore also the microstructure. The last annealing step is performed above 550°C and is a pure stress relieving treatment (8).
2.1.4 Plastic deformation and texture
The mechanisms by which plastic deformation occurs is connected to the crystal structure of a material, and depends on a number of different factors, such as number of activated slip systems and the amount of previous deformation. The number of activated slip systems is a function of loading conditions, slip planes and slip directions (11). The length of minimum slip path influences which glide system will be activated during loading (1). As mentioned titanium can consist of two morphologies (HCP and BCC) that often occur in symbiosis depending on alloy and heat treatment, each with different slip systems. In addition, grain size, grain orientation and inclusions can all be affected by plastic deformation. All these variables complicate predictions of microstructural performance during tensile loading conditions.
In addition to microstructure, texture has a strong effect on mechanical behavior (10). HCP structure is known for having strong anisotropic properties, therefore α texture has a greater influence on anisotropy than β texture (10). Figure 8 shows how tensile properties for α phase varies with loading direction, both for single crystal (a) and for textured polycrystalline material (b) and (c). The types of texture are described below.
Influence of loading direction on tensile properties. (a) Modulus of elasticity $E$ of $\alpha$ titanium single crystal as function of declination angle $\gamma$. (b) Modulus of elasticity $E$ for fully equiaxed Ti-6Al-4V ($\alpha$ grain size: 2μm) T is for transverse type of texture and B/T is for basal/transverse type of texture. (c) Yield stress $\sigma_{0.2}$ and ductility RA ($\alpha$ grain size: 2μm). All diagrams are adapted from (8).

Unidirectional hot rolling (below $\beta$-transus) of Ti-6Al-4V mainly results in two types of texture, transverse and basal type, they describe the orientation of the (0002) plane in the HCP-unit cell with reference to the deformation direction, see Figure 9a. As can be calculated from Figure 2 titanium has a c/a ratio of $\sim 1.59$. Cold rolled materials with HCP structure where the c/a ratio is lower than the ideal ratio of 1.633 tend to form texture with the basal poles tilted ±20 to 40 degrees away from the normal direction (ND) towards the transverse direction (TD), and with $[10\bar{1}0]$ poles aligned with the rolling direction (RD) (12). Temperature is a factor that influences the type of texture induced during deformation step, see Figure 9b.

[Figure 8] Influence of loading direction on tensile properties. (a) Modulus of elasticity $E$ of $\alpha$ titanium single crystal as function of declination angle $\gamma$. (b) Modulus of elasticity $E$ for fully equiaxed Ti-6Al-4V ($\alpha$ grain size: 2μm) T is for transverse type of texture and B/T is for basal/transverse type of texture. (c) Yield stress $\sigma_{0.2}$ and ductility RA ($\alpha$ grain size: 2μm). All diagrams are adapted from (8).

[Figure 9] a) shows basal (left) and transverse (right) texture schematic, with pole figures based on (0002) plane. b) shows temperature dependence of formation of texture during deformation step, for alloy Ti-6Al-4V with equiaxed structure (1).
2.2 Structure/property relations
As mentioned earlier the determination of how certain microstructural features affect mechanical properties is complicated but essential for predictions of mechanical properties of titanium.

Table 1 below lists some general trends summarizing how fine and coarse microstructure and phase arrangements influence selected properties.

Table 1 Influence of microstructure on properties of titanium alloys (1)

<table>
<thead>
<tr>
<th>fine</th>
<th>coarse</th>
<th>Property</th>
<th>lamellar</th>
<th>equiaxed</th>
</tr>
</thead>
<tbody>
<tr>
<td>○</td>
<td>○</td>
<td>Elastic modulus</td>
<td>○</td>
<td>+/- (texture)</td>
</tr>
<tr>
<td>+</td>
<td>–</td>
<td>Strength</td>
<td>–</td>
<td>+</td>
</tr>
<tr>
<td>+</td>
<td>–</td>
<td>Ductility</td>
<td>–</td>
<td>+</td>
</tr>
<tr>
<td>–</td>
<td>+</td>
<td>Fracture toughness</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>+</td>
<td>–</td>
<td>Fatigue crack initiation</td>
<td>–</td>
<td>+</td>
</tr>
<tr>
<td>–</td>
<td>+</td>
<td>Fatigue crack propagation</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>–</td>
<td>+</td>
<td>Creep strength</td>
<td>+</td>
<td>–</td>
</tr>
<tr>
<td>+</td>
<td>–</td>
<td>Superplasticity</td>
<td>–</td>
<td>+</td>
</tr>
<tr>
<td>+</td>
<td>–</td>
<td>Oxidation behavior</td>
<td>+</td>
<td>–</td>
</tr>
</tbody>
</table>

2.2.1 Regression analysis
One method to find correlations between microstructure and properties for a material is to perform regression analysis, where empirical values of properties are connected to measured microstructural parameters.

Using regression analysis based on empirical information can be successful to establish some correlations between the microstructure and properties, but the accuracy is often limited (8). Accurate predictions can only be made within the range of measured data since extrapolation beyond empirical boundary values includes a high risk of error.

Multiple regression is a common way of establishing how the summation of a number of microstructural parameters contribute to a property. The general expression is shown below in Equation 1.

\[ y = \sum W_j x_j + \theta \]  

Equation 1
The multiple regression equation relates the property $y$, such as tensile strength, to the structural parameters $x$, the weighting factor $w$, and a constant $\theta$.

In order to determine a specific $w$, one would require that the parameter $x$ associated with the $w$ is varied, while all other $x$ are held constant. In practice, holding all structural parameters fixed while varying one is impossible or very difficult (13).

### 2.2.2 Neural networks

Another method that is used for finding correlations between microstructure and mechanical properties is neural networks. A number of more detailed descriptions of neural networks and their applied uses are available in the literature (8) (14). Simplified it can be described as an adaptable computational multiple regression system, where the weighting factors are adjusted for better result. When using this method a database must be populated containing inputs such as microstructural parameters and outputs such as material properties (13). About 70% of the data is used for training the model by adjusting the weighting factors, the rest is used for testing of the model. There are different ways to train a dataset, therefore details of the model-training varies depending on what method is being used. An advantage of using neural networks is that the method makes it possible to change a single microstructural parameter while holding the others fixed (13). The use of neural networks has been used successfully to predict tensile properties for Ti-6Al-4V (15) (16).

### 2.2.3 Model parameters

Regardless of which method is used for constructing a model, the constituent microstructural parameters must be determined. Examples of parameters that have been considered relevant in other work (17) (18) (15), are size measurements of $\alpha+\beta$-grains, phase volume fraction, Widmanstätten-$\alpha$ volume fraction, grain boundary-$\alpha$ thickness, fraction of martensite-$\alpha$, $\alpha$-lath thickness and prior $\beta$-grain size. The parameters available differ between which type of microstructure being modeled and on thermo-mechanical history of material being used.

Even though no general models for describing tensile strength as a function of specific microstructural parameters exists (19), it is reasonable to believe that chances of constructing a successful model depend on both quantity and quality of structural parameters. The more information available about the materials microstructure the
more model input-parameters there are to choose from. With collection of large amounts of statistical data the accuracy of the parameters increases. To fulfill these conditions the TriBeam system can be used to gather microstructural information, making extraction of a variety of statistical information possible.

2.3 The TriBeam system
Tomography is to create a three-dimensional representation by serial sections in two dimensions. It can provide more information than a single two-dimensional image, since shapes of particles can appear different when displayed with an extra spatial dimension (20). In materials science it can be valuable to get a sense of a material’s three-dimensional appearance, like in anisotropic materials or to see heterogeneous distribution of particles or other features (21). There are several different serial sectioning techniques for materials, including; mechanical grinding, forced ion beam (FIB), atom probe tomography (APT). Lasers can also be used for serial sectioning. When employing lasers, femtosecond lasers are more suitable than conventional nanosecond- and picosecond-lasers, due to less collateral damage (22), and a smaller heat affected zone. An advantage of using femtosecond laser for material removal is that it is faster than other mentioned techniques (21).

The imaging mode(s) for a sectioning experiment depends on the type of analysis required for the material. Different imaging modes have different imaging resolution limitations and types of sensitivity (i.e. chemical sensing, grain orientation mapping). The surrounding environment also limits what is possible to use, optical microscopy can be performed in ambient air while electron microscopy require vacuum.

The TriBeam system is a recently developed serial sectioning system, which integrates several existing instruments and techniques. The unique system has been developed at the University of California Santa Barbara. It has been shown that the system can be used as a powerful material analysis instrument, especially when it comes to serial sectioning (4). Table 2 shows comparison between different types of serial sectioning methods and their removal rate, range of slice thickness, minimum imaging resolution, and the range of volumes that can be collected by the specific technique.
Table 2 Comparison between different serial sectioning (SS) methods. Atom probe tomography (APT), focused ion beam (FIB), mechanical serial sectioning (Mech-SS) and femtosecond laser serial sectioning (FSL-SS) (21)

<table>
<thead>
<tr>
<th>Method</th>
<th>Removal rate [µm³ s⁻¹]</th>
<th>Slice thickness [µm]</th>
<th>Resolution [nm]</th>
<th>Addressable volume [µm³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>APT</td>
<td>10⁻⁸</td>
<td>10⁻¹</td>
<td>0.5</td>
<td>10⁻³–10⁻⁴</td>
</tr>
<tr>
<td>FIB-SS</td>
<td>0.5</td>
<td>5–100</td>
<td>10–30</td>
<td>10⁴–10⁵</td>
</tr>
<tr>
<td>Mech-SS</td>
<td>200</td>
<td>100–2700</td>
<td>260</td>
<td>10⁵–10¹⁰</td>
</tr>
<tr>
<td>FSL-SS</td>
<td>10⁴–10⁵</td>
<td>20–150</td>
<td>260 (10–30)³¹</td>
<td>10⁷–10¹⁰</td>
</tr>
</tbody>
</table>

The TriBeam system is developed on a FEI Strata DB 235 DualBeam™ platform. The main addition is that the TriBeam system has an integrated femtosecond laser with the necessary scanning optics.

The TriBeam system is illustrated schematically below in Figure 10. A detailed description of the microscope and the detectors and their functions are described separately.

![TriBeam system diagram](image-url)

Figure 10 The TriBeam system, illustrated with its three beam sources, detectors, chamber and optical table (4)
2.3.1 Electron Beam

One of the beam sources in the TriBeam system is the electron gun, which is a component more known as a scanning electron microscope (SEM), the following section will describe the main attributes of the SEM-system.

The first SEM-instrument was developed by Knoll in 1935. Today, versions are significantly more advanced and allow visual examination of materials on a micrometer to nanometer scale. Main components in the SEM-system are the lens system, the electron gun and the electron collector (23). The electron gun provides a stable stream of electrons moving down through an evacuated microscope column. Along the column, lenses, coils and apertures that function to adjust the electron beam makes imaging possible. Figure 11a shows a typical SEM system.

The electron beam in a conventional system originate from a tungsten hairpin filament or a LaB₆ hairpin filament (thermionic emitters), while more modern electron guns are of field emission type. Thermionic emitters require heat for the electrons to overcome an energy barrier and escape towards vacuum. Field emission emitters work to overcome the energy barrier through improvements to the geometry of filament and high electric field. Field emission guns, FEGs also have the benefit of longer lifetime and better brightness. The field emission type often uses tungsten, carbon or silicon as emitting material. It consists of a wire with a sharp tip and the geometry is needle-like. The filament is negatively charged (acting as cathode) and potential is provided from a high voltage supply, 0.1-30 kV. The anode of the system is a plate with a hole in, located below the filament, where most of the electrons are captured due to ground connection.

A fraction of the electrons continues through the hole and becomes the electron beam. To channel more electrons through the hole a grid-cap (also known as Wehnelt cylinder) is placed prior the anode plate. The grid-cap has lower potential than the filament which the electron is escaping from, this results in a repulsive and therefore focusing effect. Figure 11b shows a schematic of a conventional thermionic filament and its system components. Electron lenses control the beam current by modulating the fraction of the beam which can pass through the apertures. The lenses are coils that use magnetic fields to focus the beam through the aperture holes. The reason for adjusting the beam is to obtain a suitable spot size on the sample surface. An unregulated beam would be too large to image. Most SEM can obtain a spotsize >100 nm (23). Scan coils in lower part of column control the incident position of beam on the sample surface.
Figure 11 (a) Illustration of a typical SEM column, containing electron source, lenses, apertures, deflection system and electron detector. (b) Conventional thermionic tungsten hairpin filament (23)

When the stream of electrons hits the sample surface, they will interact with the specimen and they are emitted as mainly secondary electrons and backscattered electrons. The backscattered electron are beam electrons which may penetrate the sample surface and have a changed trajectory in a elastic manner before escaping out of the surface with maintained energy. The backscattered electrons show proportional relationship with atomic number of specimen. Secondary electrons are defined as electrons escaping the sample with less kinetic energy than 50eV. The secondary electrons can originate from loosely tied outer shell electrons in the specimen, which are set to motion by collision with incoming beam electrons (23).

A detector captures the electrons and convert the electric signals into light-signals and leads them out through a vacuum-sealed quartz window. There are numerous types of detectors, attracting either secondary or backscattered electron, or both depending on type of imaging.

The beam is steered in a raster pattern over sample surface, and signals are recorded for the position of beam incident. Each position is connected to a pixel on displaying screen, giving an image of the sample surface.
2.3.2 Focused Ion Beam

The TriBeam system includes a focused ion beam (FIB) source. The basic gallium FIB-setup consists of a liquid metal ion source, ion column, detectors and a computer to run the system (24). The system has similarities to the SEM-system, which is described more detailed in the section above. Both systems require a vacuum chamber and a column with lenses, apertures and coils to control the beam.

The liquid metal ion source can provide ions of ~ 5nm in diameter (24). The liquid metal source can be of different metal types, commonly is gallium (Ga) used due to its low melting point (29.8°C) and its low volatility conserves the supply of metal and gives it a long service life (24). Figure 12 shows a schematic diagram of a typical ion source filament.

The ion emission occurs in a two-step process, first the heated Ga flows and wets the tungsten tip and an applied suppressing electric field to the tip causes the Ga to form a cone on the tip. The electrical field and the surface tension balance the cone, making its tip small enough for extracting ions. The second step is to emit ions from the cone tip with an extracting voltage, causing the ions to accelerate through the column towards the specimen surface in chamber. The current density of the extracted ions are ~1*10^8 A/cm^2 (24). The emitted ions are replaced by new due to the flow of Ga from liquid metal source. To maintain a long lifetime of the liquid metal source it should not be heated to often, usually one heat cycle keeps the metal in liquid state for several weeks. The suppressing voltage can be increased to maintain a steady flow of metal to the tungsten tip when the source ages. In a typical system the emitting electrical field is held constant and the suppressing voltage is fluctuated to obtain flow and balance of liquid metal cone.

The working distance for a FIB is much larger (~2 cm) compared to the SEM (5 mm), which allows more irregular sample geometries and surface roughness. Once the ions impact on the specimen surface several types of species are generated, such as sputtered atoms, molecules, backscattered electrons and secondary ions (24). The FIB can be used for imaging, in the same way as described for the SEM-system, where a detector records backscattered and secondary electrons. Since the ions have much more mass than electrons, low voltages are used when imaging to avoid unnecessary material removal. The penetration depth of ions into specimen surface depends on the type of material and crystallographic orientation (24).
The FIB system is often used for material removal, cutting, thinning and polishing of transmission electron microscope (TEM) specimen foils.

In the TriBeam system the ion beam can be used if necessary for fine material removal from specimen surface, in order to remove laser ablation damage, to obtain better quality of EBSD-scans.

![Schematic of liquid metal ion source in top of FIB column](24)

**Figure 12** Schematic of liquid metal ion source in top of FIB column (24)

### 2.3.3 Laser

The femtosecond laser is the source of the third beam in the system. As mentioned earlier the DualBeam™ system is supplemented with a faster material ablation method than possible with the existing ion-beam. The femtosecond laser being used is a Clark-MXR™ CPA-2110. It uses a Ti-Sapphire crystal as the primary gain medium and requires water-cooling. The pulsed femtosecond laser has an average power of 1.2 W at 1 kHz repetition rate. It outputs a 10mm diameter unfocused beam, and is focused to a beam diameter of ~5μm with a 1000mm plano-convex lens combined with a 20x long-working-distance microscope objective. The intensity of the focused beam is >10^{18} W/cm². The wavelength of the light is 780nm and the pulse width is <150 fs. Material is ablated if the laser fluence is above the materials ablation threshold, otherwise texturing and surface modification can occur. Damage and roughening of the material increases if the laser fluence is greater than ten times the threshold value (4).

The mechanisms controlling laser ablation are physically complicated, and are influenced by laser fluence, pulse duration and material properties such as melting point
and thermal conductivity (22). The free electrons of a material absorb the laser phonon energy and are heated into high temperature. The energy absorption is followed by a fast energy relaxation where the energy is transferred by thermal diffusion from the electron subsystem into the lattice. The thermal diffusion occurs through an electron-phonon coupling. The time for thermalization is in range of 1.5-3 picoseconds and increases with laser fluence (22). For a femtosecond laser the pulse duration time is shorter than the mentioned thermalization time, meaning that the laser is off when the material gets ablated. This gives smaller thermally affected zones in the material when a femtosecond laser is used, compared to longer pulse lasers such as nanosecond and picosecond lasers (25). Figure 13 below shows an example of a laser induced ablation pattern.

![Image](image_url)

**Figure 13** Femtosecond laser induced UCSB-logo, showing pulse overlap. The single laser pulses show a focused beam diameter of ~5μm (4)

### 2.3.4 Optical table

The physical dimensions of the femtosecond laser requires it to be mounted externally of the DualBeam™ chamber. It is mounted on an active air suspension dampening table, which protects the system from vibrations that might disturb the laser optics. The table is rigidly fixed to the DualBeam™ chamber, which also is air damped. The two air dampening systems are synchronized by pneumatic solenoids that are both simultaneously deflated when the sample chamber is vented. When the vacuum chamber is in use the two units are suspended uniformly.

To guide the laser beam from source to target its path goes through several mirrors and filter lenses before it enters the chamber using an optical feed-through assembly. The feed-through assembly is a laser transparent BK-7 glass window which is air sealed. On the inside of the window the final focusing lens, a 20x microscope objective is mounted.
Just before the beam enters the chamber it passes through a 45° fast steering mirror. The mirror is controlled in two axes by electromagnetic coils with fast response. The mirror steers the beam with high precision along programmed ablation patterns. The laser pulses are generally set to overlap 50% of previous pulse, both in propagation direction and in previous parallel scan line. Figure 13 seen above shows pulse overlap.

### 2.3.5 EBSD

Electron backscatter diffraction (EBSD) is a technique for performing microstructural and crystallographic analysis in a scanning electron microscope (SEM). It provides information about crystal orientation and grain characteristics from the millimeter to nanometer range. The technique directs a beam of high-energy electrons at the specimen surface with purpose of inducing backscattered electrons, as described earlier in scanning electron microscope-section. The backscattered electrons from the specimen are diffracted by the crystal lattice and detected by a EBSD-detector. The detector is a transparent phosphor screen which is energetically excited when electrons strike it (10). The screen is ~5 cm in diameter, and is held ~2 cm from specimen surface. The backscattered electrons are produced in patterns of lines or bands on the phosphor screen. The pattern occur due to the lattice diffraction, and is called Kikuchi pattern, see Figure 14a. The diffraction of electrons in lattice can be described by Bragg’s law (10).

![Figure 14](image.png)

*Figure 14* Left image (a) shows Kikuchi pattern of HCP crystal structure in Ti-6Al-4V. Right image (b) shows same image with identified bands (26)
A CCD-camera for recording the patterns is connected to the phosphor screen, either by a fiber optic bundle behind screen or by mounting the camera at a window outside specimen chamber (10). The screen usually have a thin layer of aluminum coating, which filters low energy electrons and enhances the brightness of patterns by reflecting light toward the camera, the layer also grounds the screen (10). The bands in the Kikuchi patterns are directly related to the crystal planes in the lattice. The EBSD computer software is used to interpret the Kikuchi bands and identify what crystallographic orientation they are derived from. Even though a human eye can easily identify the bands, a computer requires an advanced algorithm to do so (10).

A Hough transform is used to characterize the Kikuchi bands, it can simplified be described as follows. Boundary values are set to distinguish lighter points (pixels) in the image from background noise. Each pixel point then is assigned several straight lines bisecting it with a random direction. Each line then is assigned a perpendicular line, of which only one will go through to the Cartesian origin. The distance and angle of the perpendicular line, corresponding to its polar coordinates, is measured and stored in bins. Each point that is located along a band will have the same polar coordinates, and therefore that bin will be larger than the others, indicating that it is an interpreted band. The identified band patterns are compared to a database of known patterns from the different crystal systems and variations in their orientation to reveal what phase and orientation being imaged. The lines identified by the software are plotted over the original Kikuchi image, seen above in Figure 14b.

As the electron beam stepwise scans over the specimen surface, each interaction spot has data stored for it, such as location, orientation, phase, image quality and confidence index. Confidence index is a measurement of how reliable the interpreted bands are. The chosen stepsize, i.e. how much the electron beam is moved for each data collection, sets the boundary for how small elements the data will be stored in. To obtain good results the stepsize should be much smaller than the grain size.

For optimal pattern quality the specimen is tilted to approximately 70° tilt relative to the incident electron beam, schematic seen in Figure 15 (10). The steep specimen angle generates intense Kikuchi patterns.
2.3.6 Orientation mapping

The orientation of a crystal lattice is usually described by Euler angles, which can describe any orientation of an object in space by a sequence of rotations relative to a fixed reference system. The different grain orientations occurring in a material can be summarized and visualized by pole figures. The pole figure is derived from a projection created by poles normal to a certain crystallographic plane, inside a sphere, see Figure 16.

The pole intersects the sphere at point \( P \), where a line is drawn from \( P \) to point \( Q \), the point where the line intersects the midplane of sphere represents the two-dimensional projection of the orientation. Depicting one point per crystal or one point per datapoint will represent the complete description of poles in a polycrystalline sample. The orientations that are overrepresented in a crystal structure will generate intense fields in the projected pole figure. Another orientation description method is inverse pole figures (IPF), whereas a pole figure shows sample directions aligned with a particular crystallographic pole, an IPF does the opposite, indicating the poles aligned with a
specific sample direction (10). *Inverse pole figures and pole figures* can be seen further down in the result section as Figure 34 and Figure 35 respectively.

### 2.3.7 EDS

EDS or energy dispersive spectroscopy-d detector is used for chemical analysis of materials. The detector works together with an electron or ion beam. The beam is focused on the sample surface and hits the top layer of atoms. The atoms are originally in its ground state, with its surrounding electrons shells. The beam impact may excite some electrons from its origin shell, creating an electron hole. Outer shell electrons that have a higher state of energy might decay to fill the hole. The difference in energy is then emitted as an X-ray. The energy difference between shells gives different wavelength of X-rays. The EDS-detector registers energy and number of the emitted X-rays, by converting incoming X-ray photons to photoelectrons and voltage pulses. Each voltage pulse is proportional to the energy of incoming X-ray photon (23). The detected information gives a chart with peaks for each recognized element and its structure. The EDS in TriBeam is a peltier-cooled EDAX Apollo device.

### 2.3.8 Custom stage

The DualBeam™ has a five axis positioning stage, but with the laser optics installed the positioning necessary for laser ablation is limited. Therefore, to obtain perfect alignment of sample with the beam being used, a custom made microstage has been manufactured. The microstage is mounted as a separate unit on top of the existing DualBeam positioning stage. Its main purpose is to hold the sample and move it to accurate position with respect to the instrument being used. The beam spot on the sample surface has to be kept in eucentric position for all tilt angles. The beam sources are all fixed so their beams coincide at a working distance of 5 mm from the pole piece. The sample has to be aligned with great precision to obtain eucentric position, otherwise the imaging area or the material being removed may be incorrect.

The microstage has Attocube™ piezoelectric driven positioners, one per axis in x, y, z-direction and tilt. After a layer of material has been ablated, the exposed surface will be lower than the eucentric height. To compensate for the ablation, the sample must be raised in z-direction by adjusting the z-positioner the same distance as the thickness of the layer of ablated material. This ensures that the freshly laser machined surface always is located at the eucentric position before a new cycle is started.
When ablating with the laser, the ablated material has to re-deposit elsewhere. It sputters ballistically and condenses on favorable surfaces that have linear paths to the original ablation location. To avoid re-deposition of material on sensitive instruments inside the TriBeam chamber, a shutter has been developed. The shutter works as a shield around the sample while it is being laser ablated. Material re-deposit on the inside of shield thereby protecting pole piece, electron gun and other surfaces. The shutter is mounted on the microscope stage and is inserted and extracted automatically each time the laser is being used. Figure 17 shows the microstage schematic and in-situ.

Figure 17 (a) shows schematic of custom made microstage, (b) shows microstage in-situ in the Tribeam, tilted to 70° for EBSD data collection (4)

2.4 3D-data collection and reconstruction
As described earlier, three-dimensional datasets are mainly collected by serial sectioning which is a destructive sampling technique. An alternative is to use X-ray methods, which are non-destructive techniques, leaving the sample intact and therefore allows time-dependent studies of microstructure during thermal or mechanical input (10). X-ray methods require high intensity X-ray sources to penetrate through high atomic number elements (10). For structural materials, X-ray tomography often does not provide suitable contrast for the differentiation of phases with similar densities. As such, dense materials are typically serial sectioned using destructive techniques. Serial sectioning techniques consist of two main steps, sectioning and imaging, which is iteratively repeated until the desired sample volume is collected.
The reconstruction of three-dimensional data starts with mounting all the two-dimensional images into a three-dimensional array. The slices are aligned on top of each other, by the defined slice spacing. The spacing corresponds to the thickness of the material that was ablated during the sectioning experiment. The pixels are elongated in z-direction, i.e. the normal of the pixel plane, to fill the space to the next slice and create a solid model. The pixels are now transformed into voxels, which still contain the same information. The reconstruction software outputs raw voxelized data that can be viewed or processed by other programs.

Each raw data point contains information about each voxel such as position, phase, orientation, confidence index and segmented grain data. The voxels may have the shape of an elongated cube, often stretched in the direction normal to sectioning because of greater imaging resolution compared with sectioning slice thickness. Each grain has a grain identification number, a set of spatial coordinates and a known volume derived from the smaller voxel elements.

Reconstruction of three-dimensional datasets from two-dimensional slices can be made using a variety of different software packages. Here reconstruction was performed using the DREAM.3D software. When performing reconstruction using the DREAM.3D software, where it is possible to choose different grain segmentations based on, misorientation tolerance angle and minimum allowed grain size. The misorientation tolerance angle (MTA) determines how large of a difference in orientation is used to segment voxels into discrete grains, where the orientation is described by Euler angles. A minimum allowed grain size filter was used, which determines the minimum number of voxels a grain must contain, in order to be classified as a grain.

Changing the misorientation tolerance angle and the minimum allowed grain size filter modifies the definition of a grain, and microstructural statistics.

In the DREAM.3D software, the grain segmentation starts by identifying seed voxels, which are voxels of high quality where the grains will "nucleate" and grow as new voxels are added. Adjacent voxels are compared to the seed voxel and if their orientation falls within the defined tolerance (MTA) they are included to the grain being segmented. The grain growth continues by comparing all voxels to their neighbors, determining if they belong to the same grain. It is important that the voxels are compared both to their nearest neighbors and the seed voxel, otherwise the orientation difference between grain center and periphery could be larger than the desired MTA-value. An example of
this is seen in Figure 18 where the misorientation between the grey seed voxel and red voxel could theoretically be 7 degrees, which would exceed a MTA value of five.

![Figure 18](image)

**Figure 18** Schematic of possible accumulated misorientation that could occur if misorientation tolerance angle (MTA) would be set to five degrees and if voxels are only compared with nearest neighbors-voxels and not to grey seed voxel (10)

The same conditions are used when performing analysis of single EBSD scans, in which case each datapoint is two-dimensional, and data is stored in pixels instead of voxels. In the two-dimensional EBSD OIM Analysis software (EDAX), the default setting for MTA is equal to five, and minimum grain size equal to two. Data points (either pixels or voxels) with poor image quality can be weighed against neighboring grain values, to modify these data points which may be erroneous. This data cleaning is performed by software algorithms, which compare each data point to its neighboring data points, and modify the data according to the kernel. Figure 19 below shows a cleaning example in two-dimensions.

![Figure 19](image)

**Figure 19** Illustration of 2D cleaning of data points with poor quality

Data cleaning in three-dimensions is more reliable than in two-dimensions, because in three-dimensional datasets voxels are surrounded by additional layers of voxels above and below it, offering more neighbors for comparison.

### 3 Method

The experimental methods will be described in three sections, one focusing on the material preparation, one describing the TriBeam operation conditions, and one outlining the data structure.
3.1 Sample preparation and heat treatment
The as-received material was wire electrical discharge machine (EDM) cut from forged plates into bars with cross section measuring 13,48 x 12,69mm. The rectangular bars were sectioned into samples as shown in Figure 20, using a liquid cooled high-speed diamond cut-off saw. The samples measured approximately 3,5 x 1,4 x 13,5mm.

![Figure 20 Illustration of the as-received material being cut into samples](image)

Six samples were heat-treated. Two types of heat treatment were performed, which will be referred to as HT1 and HT2. The heat treatment schedules for HT1 and HT2 shown in Figure 21 and Figure 22, respectively. Time and temperature were chosen to obtain two similar microstructures both having low amounts of accumulated plastic damage.

![Figure 21 Time and temperature diagram for Heat treatment 1 (HT1)](image)
After heat treatment the samples were mechanically ground and polished to obtain a near 90° edge, to facilitate laser beam alignment and milling in the TriBeam. Grinding was performed using 600, 800, 1200 grit SiC paper followed by polishing with 6μm diamond solution. A final vibratory polishing step in colloidal silica solution was performed for 24-48 hours to samples that were intended for observation in SEM. A sample of the as-received material was also metallographically prepared with the same conditions as the other heat-treated samples.

Electro polishing was also performed as an alternative to the vibratory polishing step, with etching times between 12 and 35 seconds with potential range between 15V and 25V were used. The electro polishing solution contained 6vol% Perchloric acid, 35vol% Butoxyethanol and 59vol% Methanol.

One piece of the HT1 sample was later ground to a thickness of 600μm, and then wire electrical discharging machine (EDM) cut into seven freestanding pedestals. The pedestal sample is illustrated in Figure 23. The top surfaces of pedestals measured approximately 600x600μm. Optical images of pedestal-sample were collected for verification of the geometry.

Figure 23 Illustration of Electric Discharge Machine (EDM) pedestals, cut from on HT1 specimen
3.2 TriBeam settings
The settings used in TriBeam instruments during data collection are presented in Table 3, the time for serial sectioning steps in data collection cycle is presented in Figure 24.

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Figure 24 Diagram showing the time for serial sectioning steps in data collection cycle, total time per cycle was 42 minutes. EBSD and ion milling are clearly the rate limiting steps.

3.3 Data handling
The EBSD data was obtained as individual scans for each slice. The DREAM.3D software was used for reconstructing all the gathered two-dimensional slices into one three-dimensional dataset, and for outputting data containing grain information such as; grain ID, volume, voxel positions and phase. The grain information data was imported and processed using MATLAB code to extract more specific microstructural statistics described in section below. For visualization of three-dimensional models the software Paraview was used. Figure 25 shows a flowchart of the data handling process.
MATLAB code was developed to handle large three dimensional arrays of reconstructed data and to extract statistical information from the dataset. The datasets are stored in MATLAB in three-dimensional matrices, where the value of each element corresponds to a voxel, which stores the grain ID number that has been assigned during the reconstruction and segmentation process. This makes it possible to manipulate or modify the matrix and extract statistics from the voxels included in a specific sectioning slice or to analyze the total volume of dataset. Also it is possible to extract three-dimensional statistics from all grains that are bisected by a plane, giving “pseudo-3D” information, this is illustrated in Figure 46. Using the DREAM.3D software additional information is available for each grain, such as type of phase and volume. This information is also imported into MATLAB and linked to their corresponding matrix elements. The MATLAB code has a built-in size filter to exclude grains smaller or larger than desired volume, or alternatively, exclude specific individual grains entirely from statistical extraction. Grains that touch edge of the dataset are referred to as edge-biased grains and are by the code automatically excluded from statistical calculations.
Figure 25 Flowchart illustrating the data handling/processing and software tools being used for data collection and 3D-reconstruction

4 Results and Discussion

The obtained results and discussion are integrated and presented as subdivided sections with description of encountered problems and their solutions, in chronological order.

4.1.1 Microstructural Characterization
Backscattered electron images were taken using SEM, of the samples HT1, HT2 and for the as-received material, in order to determine whether changes in the microstructures occurred following heat treatments. The HT1 and HT2 samples seen in Figure 26 shows a recrystallized fully equiaxed microstructure, with β grains located at α grain triple-points. The HT2 sample has slightly larger grains than HT1, also the β phase in HT2 is less globular than in HT1, due to the higher temperature and longer heat-treatment.
Figure 26 Back scattered electron images of heat-treated samples. (a) HT1 sample, (b) HT2 sample and (c) is the as-received material

4.1.2 Data collection
Data collection with the TriBeam system requires the optimization of some parameters for each sample and material, such as laser alignment, laser fluence and SEM imaging instrument settings. Several data collection attempts were performed, which revealed factors that might interfere with the quality of data collection. For alloy Ti-6Al-4V it is necessary to “clean” the top layer of surface after laser ablation using focused ion beam (FIB). The laser ablation damages the surface, resulting in poor EBSD scan quality. Different ion beam angles and milling depths for FIB polishing were tried. Excessive ion beam milling depth resulted in damaged surfaces and preferential material removal visible in Figure 27. Shallow depths of ion milling resulted in little to no increase in EBSD pattern quality.

Figure 27 SEM image of sample surface after excessive Focused Ion Beaming (FIB)
Optimal FIB-settings were established not only to obtain high quality EBSD patterns, but also for minimizing the time for each data collection cycle, as could be seen in Figure 24, the steps of FIB milling and EBSD imaging has a considerable influence on data collection cycle time.

Another factor limiting both quality and size of collected dataset is re-deposition of ablated material. Depending on depth of the laser ablated surface, the re-deposited material forms on the walls surrounding data collection area. Figure 28a shows a SEM image of a laser machined and FIB polished area, prepared for data collection, with re-deposition on the nearby edges. Image insets, b and c in Figure 28 show EBSD scans colored by image quality and IPF, respectively. Figure 29 shows the same sample 149 data collection-cycles later, wherein the re-deposition walls have now grown significantly larger and result in poor EBSD quality, shown in b and c.

**Figure 28** Image (a) shows sample after several laser ablation iterations and after focused ion beaming (FIB). Images (b) and (c) shows EBSD scan of the FIB-area, colored by image quality and orientation (IPF) respectively.
Figure 29 Same imaging as in Figure 28, seen 149 data collection cycles later. EBSD scans have low quality due to shadowing from re-deposited material.

Figure 30 shows a detailed image of laser ablation induced re-deposition of the sample material.

Figure 30 High magnification SEM image of re-deposited material after laser ablation.
The walls of re-deposited material interfere with the trajectory of backscattered electrons going from specimen to EBSD detector, preventing proper indexing of Kikuchi patterns by means of the Hough-transform. Figure 31 shows a Kikuchi pattern with shadowing due to re-deposited material.

Figure 31 Kikuchi pattern from EBSD scan, the black fields are due to shadowing from re-deposition walls, interfering the Hough-transform band interpretation

4.1.3 Pedestal sample geometry
To obtain larger and higher quality datasets a pedestal sample was fabricated, see Figure 32. The motivation for the pedestal was to eliminate nearby edges for ablated material to condense on. This requires that pedestal measurements do not exceed the maximum laser scan range. Figure 33a and b show pedestal in TriBeam before and after laser ablation, notice that there is no visible re-deposition of ablated material. Unfortunately, no complete dataset could be acquired using this type of sample geometry within the time limits for this project, due to EBSD-detector failure.

Figure 32 Optical micrograph of the pedestal sample made from material HT1. The pedestals were cut using a wire electrical discharging machine (EDM)
Figure 33 Left image (a) shows an SEM-image of a pedestal in TriBeam before laser ablation. Right image (b) shows the same pedestal from (a) after laser ablation and FIB milling

4.1.4 Collected dataset
The TriBeam system acquired the three-dimensional dataset in shorter time compared to other available sectioning methods. The total collection time for data acquisition was 37 times faster compared to if the FIB had been used for the serial sectioning step.

The three-dimensional model obtained from the reconstruction of the collected Ti-6Al-4V dataset (not from pedestal sample) is seen in Figure 34. The dataset measured 106x241x190 μm with an imaging resolution of 0.6 μm and a sectioning thickness per slice of 1 μm. The color scheme is the same as seen in OIM software, corresponding to inverse pole figure (IPF) colors. Each color corresponds to the orientation shown in the IPF-map in Figure 34.
Figure 34 Three-dimensional reconstruction of dataset, alloy Ti-6Al-4V. IPF-colors are applied with reference to RD direction, and can be interpreted with the inset IPF-map.

A textured region of grains is visible in the microstructure which all have IPF orientation near the [10 10] direction (mapped in blue color). To establish the type of texture, three different EBSD images from the dataset were analyzed to obtain pole figures. The three slices taken from top, middle and bottom section of dataset corresponds to a total number of 1936 orientation-analyzed grains. Obtained pole figures are seen in Figure 35.
Figure 35 (a) Pole figure for α-phase, showing two red “hot-spots”, indicating mainly transverse type of texture. (b) showing texture for β phase. The (110) reference plane corresponds due to cubic symmetry to the (101) plane, seen in IPF-map in Figure 34.

The two red areas in Figure 35 indicate that α phase texture is mainly of transverse type, which means that the basal pole is aligned with the transverse direction (TD). Also some intensity fields are seen at the RD region, by comparison to the two right hand side examples in Figure 9b, it is likely that the hot rolling was performed below, but near the β-transus temperature.

The texture occurs due to the deformation step and the activated slip system is mainly of prismatic type. When performing deformation at high temperature in the α+β phase field the volume fraction of β phase is high, and a β phase texture occurs (8). In the subsequent cooling sequence β phase transform into α phase, the orientation relationship between the parent β phase and the formed α phase is fulfilled by (110)β||{0001}α (8).

It has been proposed that the β texture occurs due to its ability to accommodate the rolling deformation (27), also it has been shown that nucleation of new β grains are thermodynamically less favorable than growth of existing β phase during heating (28). To fully understand the influence of deformation on the β texture (and indirect the subsequent α texture) would require orientation information obtained prior deformation step. Figure 43 further down shows the β grains visualized for different volume intervals, it shows that the β phase has a preferred orientation described by the green IPF-color. The green color corresponds to the (101) as reference plane, which is symmetrically equivalent to the (110) plane.

4.1.5 Grain segmentation
The Ti-6Al-4V dataset was initially using misorientation tolerance angle (MTA) of five degrees, or greater, and minimum grain size set to eight voxels. The diagram in Figure
36 shows grain volume for each of the $\alpha$-grains and the presence of a few significant outliers.

![Diagram showing the grain volume for each $\alpha$ grain](image)

**Figure 36** Diagram showing the grain volume for each $\alpha$ grain, which shows that some grains are larger than the average. The volume of the largest grain is approximately $1 \times 10^6 \, \mu m^3$. The smaller inserted diagram shows volume range from $0-3 \times 10^5$ voxels

The large grains from Figure 36 were identified and visualized to determine the nature of their abnormal sizes this is shown in Figure 37. The top left image, denoted a) shows the whole dataset colored by IPF-colors, while b) shows same dataset colored by grain-ID, where each grain has unique color. The visualization in Figure 37b shows segmented grains that are larger than average. Figure 37c shows the three largest grains in the dataset, colored by IPF-colors. Figure 37c shows more explicitly that the voxels belonging to the three abnormally large $\alpha$ grains could be subdivided into smaller grains, indicating that the MTA value is incorrect. The difference in orientation of these grains is obviously small, which will be discussed further. These three large grain clusters were not appropriately subdivided into individual grains because the MTA-value used for grain segmentation was set too large.
Figure 37 Grain segmentation using MTA 5, image (a) is colored by IPF-colors while image (b) is colored by grain ID. Image (c) shows the three largest grains, identified as clusters.

The MTA values were varied to get the DREAM.3D software to output a more realistic dataset, without α phase grain clusters. The diagram in Figure 38 shows how the grain volume and number of grains vary with MTA.
Diagram showing how MTA affects number and volume of α and β phase respectively in 3D-reconstruction

The number of β phase grains decrease by less than 5% for MTA 2°-5°. The volume of the β grains remains nearly constant for all the shown values of MTA. The number of α phase grains varies linearly, adding roughly 200 grains per 1° increment in MTA between 5° and 2°. Between 2° and 1° MTA the number of grains increase dramatically, by almost 300%, showing that the larger α grain clusters have been subdivided.

To better understand differences in segmentations using MTA of 2 and 1° for α grains, the largest clusters for these reconstruction versions were visualized. These visualizations for α grains are shown in Figure 39, which also shows that a large α grain cluster in Figure 39b (MTA = 2°) has decreased in size compared to Figure 39a (MTA = 1°). Conclusion from this is that using a MTA of 1°, instead of 2°, divides the single cluster seen in Figure 39b into the four smaller clusters seen in Figure 39a. Also the volume of clusters in Figure 39a, is less than in Figure 39b. The observed volume decrease was investigated to reveal how the subdivided voxels were segmented.
**Figure 39** Left image (a) shows four α clusters in textured region. Right image (b) shows one single α cluster, a and b is for misorientation tolerance angle (MTA) 1° and 2° respectively.

The image table in Figure 40a-i, shows visualizations of grains with volumes contained in the specified range as a function of MTAs. Comparison of Figure 40d with Figure 40a reveals a change in the distribution of small grains. A significant fraction of the grain clusters in Figure 39b are subdivided into smaller grain fragments when using MTA of 1°. Each fragment is now considered as a discrete grain by the DREAM.3D software. Visual comparison of the size and shape of the fragments with the grains shown in Figure 40d and Figure 40g demonstrate that it is unlikely that they are real individual grains, but more likely an artifact caused by the low MTA value.
The quantity and size of these fragments would have a significant impact on grain volume statistics. To obtain more accurate statistical results one option would be to exclude the fragments using the size filter in the MATLAB code, another option is to change the minimum allowed grain size-function in DREAM.3D software during

Figure 40 α grains visualized for different volume ranges and misorientation tolerance angles (MTA). Volume is in voxels and MTA values are in degrees.
reconstruction. The benefit of the later approach is that more data is kept instead of excluded. It is desired to use as much of the collected data as possible, to obtain a realistic model. The data points in the fragments do not have low diffraction image quality, but the segmentation has failed to index them into realistic grains.

By using the *minimum allowed size*-function the fragments are forced to join adjacent neighboring grains, instead of being excluded. The function can be applied for each phase separately, or for an entire dataset. The MTA grain segmentation for this dataset does a poor job defining the α grain boundaries, but is able to distinguish β grains, which are shown further down in Figure 43, therefore the *minimum allowed grain size*-function was only applied to the α phase voxels.

To determine the *minimum allowed grain size* to use for the α phase Figure 40 was analyzed to determine when typical α grain shapes emerged. The minimum allowed grain size was therefore set for volumes less than 124 voxels (4.4 μm equivalent diameter). Grains smaller than 124 voxels show an irregular shape, often consisting only of a few voxels. Further TEM-experiments would be useful to determine if the small grains are real or if they are improperly segmented grains. The imaging stepsize used for the EBSD scans was 0.6 μm, which is also a limitation, since it determines minimum size of a data point and limits the number of properly indexed pixels available to characterize a grain.

Finalized reconstruction of the dataset were made using *minimum allowed grain size* set to 125 voxels for α phase and 8 voxels for β phase and manually removing the remaining α grain clusters seen in Figure 41. Visualizations of the finalized datasets are shown in Figure 42. The grains have been segmented and the remaining fragments from Figure 40a no longer exist.
Figure 41 Visualization of the identified and excluded α grain clusters from the dataset with segmentation settings of MTA 1 and a minimum allowed α grain size of 125 voxels. Left image (a) is colored by unique grain ID while right image (b) is colored by IPF-colors.

Figure 42 Rendering of the remaining α grains after grain clusters were removed, using segmentation parameters of MTA 1 and minimum allowed α grain size 125 voxels. The grains have an expected shape and grain fragments (as seen in Figure 40a) no longer exists. The left image a shows grains with volume ranging between 125-499 voxels, while the right image b shows grains with volumes between 500-1454 voxels.
4.1.6 Verification of grain segmentation

To verify if previously mentioned reconstruction settings and adjustments resulted in an accurately segmented dataset, a comparison to a two-dimensional cross-sectioned sample was performed. An EBSD dataset was collected from the same HT2 sample material that the 3D-dataset was obtained from, and is shown in Figure 44. This EBSD data was collected from a metallographically prepared specimen that was vibratory polished in colloidal silica and then imaged with a stepsize of 0.4 μm. The dashed box shown in Figure 44 is a region of equal sized area and similarly textured as the slices gathered during the TriBeam serial sectioning experiment. The inset EBSD dataset area was cropped and analyzed in OIM software in order to compare it with data from the TriBeam serial sectioning experiment.
Average grain area and phase fractions were compared between the serial sectioned EBSD slices and the large reference two-dimensional EBSD area collected and shown in Figure 44. The OIM software calculates an “equivalent area”, which is the two-dimensional grain area corresponding to the area of an ellipsoidal fit. The compared grain area results for both α and β phases are shown below in Table 4.

<table>
<thead>
<tr>
<th>Average Grain Area</th>
<th>Alpha (phase fraction)</th>
<th>Beta (phase fraction)</th>
</tr>
</thead>
<tbody>
<tr>
<td>All 2D slices in 3D-dataset</td>
<td>61.48 μm² (0.92)</td>
<td>7.02 μm² (0.08)</td>
</tr>
<tr>
<td>Reference scan entire area</td>
<td>59.52 μm² (0.96)</td>
<td>4.9 μm² (0.04)</td>
</tr>
<tr>
<td>Reference scan boxed area</td>
<td>59.74 μm² (0.97)</td>
<td>5.0 μm² (0.03)</td>
</tr>
</tbody>
</table>

It should be mentioned that the values from the two samples may not be directly comparable because the reference scan is a single EBSD scan while the three-
dimensional dataset is represented as the average of multiple slices. Also, the calculation of “equivalent area” by the OIM EBSD software is not equivalent to grain area measured from the three-dimensional dataset, which is the summation of the pixels associated with a grain. Since a cross section of a grain may not perfectly fit by an ellipse, the measurement of area based on number of pixels should be more accurate, especially for β grains that have a less equiaxed shape. Despite these reasons, the similarity of the values in Table 3 indicates that the grain segmentation parameters selected for the three-dimensional dataset give a reasonable result.

To further investigate the quality of the EBSD grain segmentation, the α grain volume was converted to equivalent sphere radius (ESR) and normalized by its average radius (ESR/<ESR>) and plotted as a histogram showing grain size distribution. The histogram was plotted versus theoretical distribution curves, as shown in Figure 45.

![Grain Size Distribution](image)

**Figure 45** Grain size distribution for the α phase in the reconstructed 3D-dataset, compared to the theoretical distribution curves. The horizontal axis shows volume converted to equivalent sphere radius (ESR) and normalized by the average equivalent sphere radius (<ESR>), equal to 5.55 μm. The vertical axis shows the grain fraction.

The grain size distribution in Figure 45, shows that the grain size distribution fit well by the log-normal curve, proposed by Feltham (29), which has been used to fit grain size distributions in nickel and iron (30) (31).

The log-normal expression is seen in Equation. 2
\[ f(R) = \frac{b}{\sqrt{\pi} R} \exp \left[ -b^2 \left( \ln \left( \ln \frac{R}{R_m} \right) \right)^2 \right] \]  

Equation. 2

where \( R_m \) is the median value in the log-normal distribution of ESR/<ESR>, and was determined to be 0.94 for the titanium dataset. The constant \( b \) was calculated to be 1.77 by least-squares method. The distribution suggested by Hillert (32), is based on a combination of Ostwald ripening and grain growth and is described in Equation 3.

\[ f(u) = A \frac{u}{(2-u)^{2+\beta}} \exp \left( -\frac{2\beta}{2-u} \right) \]  

Equation 3

where \( u=R/R_{cr} \) and \( R_{cr} \) are given by \(<R>=8R_{cr}/9\). The index \( R \) is radius and \(<R>\) is the average radius of grains in dataset. For three-dimensional growth the constant \( \beta \) equals 3. The constant \( A \) was calculated by least-squares method to have a value of 41. Another distribution function was suggested by Louat (33), which assumes that grain boundary movement is similar to a diffusion-like process (31). The function is given by Equation 4

\[ f(R) = C R \exp(-aR^2) \]  

Equation 4

where constants \( C \) and \( a \) were determined to be 0.4 and 1.08 respectively, by least-squares method. Figure 45 shows that the Hillert function predicts that the highest fraction of grains should be represented by grains with volume slightly larger than the average grain volume. In comparison to the function suggested by Louat, which predicts a lower distribution peak and a larger number of smaller volume grains, the Hillert function has a shorter tail but does not overestimate the number of small grains as much. The good fit of the log-normal curve to the three-dimensional dataset indicates that the distribution of grains follows classical models.

4.1.7 Statistical extraction

Extracting statistics from three-dimensional dataset offers the possibility to use conventional methods, i.e. two dimensional slicing but with the benefit of being able to choose any direction as normal to sectioned plane, see Figure 46a. Also non-conventional types of sectioning can be performed since an additional spatial dimension is available. Figure 46b shows an example of such a method, referred to as “pseudo-3D”,

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where three-dimensional information is gathered from all grains being intersected by a two-dimensional plane.

**Figure 46** Left image (a) shows a 2D slice from dataset while right image (b) shows same slice in “pseudo-3D” which contains three-dimensional information of all grains intersected by two-dimensional plane.

The average grain volume for all the grains intersected by a plane transversing through the dataset was plotted as a function of position of the plane, with RD, TD and ND-direction as the normal faces to the plane. It was found that α grains in textured regions have smaller grain volumes compared to α grains in the surrounding bulk structure. The average volume for β grains was found to remain uniform through the dataset regardless of structure, see Figure 47. The median value of the grain volumes followed the same trend as the curves seen in Figure 47.

**Figure 47** Pseudo-3D grain volume data for slices with ND normal to the intersection plane. The red and blue curves represent α and β phase respectively. The average α grain volume is shown to be smaller in textured region of the dataset, when compared to bulk.
Similar to the average volume of α grains, the same trend was observed for 2D-slices plotted as a function of position, where the average grain area was seen to be smaller in the textured regions. Additional figures of two- and three-dimensional measurements can be seen in the appendix.

The number of grains together with microstructural parameters and neighboring grain information extracted from the dataset are presented in Table 5 and Table 6.

**Table 5 Amount of grains in dataset and extracted volume and fraction parameters**

<table>
<thead>
<tr>
<th>Total No of grains</th>
<th>No. of grains used for statistics</th>
<th>No. α grains</th>
<th>No. of β grains</th>
<th>Volume fraction α phase</th>
<th>Volume fraction β phase</th>
<th>Grain fraction α phase</th>
<th>Grain fraction β phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>8893</td>
<td>6645</td>
<td>2484</td>
<td>4161</td>
<td>0.9353</td>
<td>0.0647</td>
<td>0.3738</td>
<td>0.6262</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Average grain volume α [μm³]</th>
<th>Average grain volume β [μm³]</th>
<th>Median grain volume α [μm³]</th>
<th>Median grain volume β [μm³]</th>
<th>Percent used voxels in dataset</th>
</tr>
</thead>
<tbody>
<tr>
<td>1043</td>
<td>43</td>
<td>599</td>
<td>17</td>
<td>57</td>
</tr>
</tbody>
</table>

**Table 6 Type and average amount of neighboring grains for α and β grains in dataset**

<table>
<thead>
<tr>
<th></th>
<th>α+β grains</th>
<th>α grains</th>
<th>β grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>α neighbors</td>
<td>8.1</td>
<td>12.6</td>
<td>5.4</td>
</tr>
<tr>
<td>β neighbors</td>
<td>3.3</td>
<td>6.9</td>
<td>1.1</td>
</tr>
<tr>
<td>α+β neighbors</td>
<td>11.3</td>
<td>19.5</td>
<td>6.5</td>
</tr>
</tbody>
</table>

The distribution of contiguos neighbors for both phases in dataset is shown in Figure 48. The value of <11.3> for this dataset deviates from the value 14, which is the one of a tetrakaidecahedron shaped grain, suggested by Kelvin (34), but is near what has been observed and presented based on experiments of other metallic alloys (31), such as for nickel <12.9>, β-brass <11.8>, Al-Sn alloys<12.48> and for iron<12.1>.  

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5 Conclusions

By using the TriBeam system to collect a three-dimensional dataset of alloy Ti-6Al-4V and by reconstructing and analyzing the obtained dataset, the following conclusions were drawn.

The TriBeam permitted acquisition of a large three-dimensional dataset from Ti-6Al-4V with grain orientation information. Characteristics of the collected dataset include:

- Dataset volume = 190x241x106 μm
- Dataset acquisition time = 75h
- Dataset was gathered in a fully automated scripting

- β and α grains were reconstructed in three dimensions showing their morphology and connectivities
- The α grain size was volumetrically smaller in highly textured regions, compared to the bulk structure, while β grain volume remained evenly distributed through structure
• Dataset size and EBSD imaging quality of dataset can be improved by modifying sample geometry into micropillar specimens

• Highly textured microstructure zones were shown to have a high density of α grain boundaries with misorientations between, or less than 1° to 2°

• Grain volume distributions for collected EBSD datasets are well represented by a log-normal distribution proposed by Feltham
6 Proposed future work

As a proposal for future work in succeeding projects, some topics of interest are discussed.

6.1 Future statistics

Further development of the MATLAB code could yield the extraction of additional microstructural parameters from the three-dimensional dataset. Examples of parameters that could be measured and quantified are grain shape descriptors, variability and texture of grain orientation, egde-to-edge distance for β grains and grain boundary surface areas. These parameters are described below.

6.1.1 Grain shape and grain boundary distribution

Accurately describing grain shape is complicated since each grain has individual features and therefore cannot be assumed to have a generalized shape. One method is to fit a tri-axial ellipsoidal body around a grain, which would not describe a fully dense microstructure, but could give a close approximation of the grain shape. The three radii in the ellipsoid could be derived and quantified from the voxel position information and connected to the ellipsoidal volume, to reveal if such a shape could be used to describe the grains.

The grain boundaries within the dataset could be characterized in order to map distribution of internal interfaces, this could be of interest since it has been shown that grain boundary engineering can improve properties such as sliding, cavitation, corrosion and fracture (35). To distinguish grain boundary types, the values of five different parameters must be determined (36). Three of these parameters describe lattice misorientation (e.g. Eulerian angles) between neighboring grains, and the two remaining parameters (spherical angles) are needed to determine the interface normal. Two-dimensional EBSD data can be used to determine four of these parameters, but the fifth is an angle representing the surface normal out of the plane and require three-dimensional information (36). Characterization of internal interfaces could be performed in three-dimensional dataset, since orientation and position of voxels located at grain boundaries can be extracted.
6.1.2 Surface area and grain smoothening
The surface area of grains in the dataset could be measured on grains in their present shape, but due to the voxel-shape all surfaces have a jagged shape, the calculated surface area would therefore not be accurate. In order to obtain a more even surface, the grains must be smoothened by software. Smoothening was performed on dataset, but result was not satisfactory due to the range in grain size. Small grains with less than three neighbors lack grain boundaries with triple and quadruple points. The smoothening algorithm that was used iteratively oscillates the nodes of a meshed grain surface, while the triple and quadruple points are held fixed, until a more even surface is obtained. In the dataset the smoothening resulted in that small grains were reshaped into thin discs. The three-dimensional dataset collected in this project would require another type of smoothening algorithm, in order to obtain accurate surface area information. Surface area could be used for calculating surface to volume ratio.

6.2 Heat-affected zone
When using EDM-cut micropillar specimens for data collection, it is desired to collect data from microstructure that has not been affected by the heat evolved during the fabrication step. To avoid this, the heat-affected zone due to EDM cutting could be determined for Ti-6Al-4V.

6.3 Characterization of Ti-6Al-4V lamellar microstructure
A significant part of the work that has been published about Ti-6Al-4V and its structure-properties are based on two-dimensional experiments on microstructure containing lamellae. By producing and characterizing Ti-6Al-4V samples with lamellar microstructure (fully lamellar or bimodal), comparison to more literature would be possible. To obtain good resolution of lamellar structure would require that EBSD stepsize and ablation depth be selected with precision.

6.4 Texture occurrence
It is known that the texture in Ti-6Al-4V has great influence on tensile properties. Therefore it would be of interest to quantify to what extent the microstructure is textured. Since it has been shown that α grains have smaller volume in textured region compared to bulk structure, it could affect grain statistics extracted for use in future models.
7 References

5. Dawson P, Pollock T, Miller M, Williams J. Development of Bounds of Strength and Ductility of Titanium Alloys Produced by Traditional and Low-Cost Methods Based on Explicit Linking of Microstructures to Properties..
34. Thompson (Lord Kelvin) W. On the division of space with minimum partitional area. Acta Mathematica. 1887.
8 Appendix

Figure A1 Pole figures showing β-phase texture for orientations (001) and (111)

Figure A2 Pseudo-3D grain volume data for slices with TD normal to the intersection plane. The green and blue curves represent α-phase and β-phase respectively. The average grain volume for both phases is evenly distributed through the dataset. The peak value seen to left part of the green curve is due to one single grain that was intersected by plane.
Figure A3 Pseudo-3D grain volume data for slices with RD normal to the intersection plane. The purple and blue curves represent α-phase and β-phase respectively.

Figure A4 Average cross-sectional grain area as a function of position in dataset, with ND as normal to sectioning plane. The average grain area is smaller in textured regions compared to surrounding bulk.
Figure A5 Average cross-sectional grain area as a function of position in dataset, with TD as normal to sectioning plane. Top green curve represents α-phase while bottom blue curve represents β-phase.

Figure A6 Average cross-sectional grain area as a function of position in dataset, with RD as normal to sectioning plane. Top purple curve represents α-phase while bottom blue curve represents β-phase.

Figure A7 Area fraction of phases as a function of position in dataset, ND as normal to intersection plane. A decrease of α-phase fraction can be seen in textured region.