INVESTIGATION OF Ti6Al4V MICROSTRUCTURE EFFECTS ON PROCESS MATERIAL INTERACTION DURING MICRO MILLING

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Investigation of Ti6Al4V microstructure effects on process material interaction during micro milling
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December 2017

We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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ABSTRACT

INVESTIGATION OF Ti6Al4V MICROSTRUCTURE EFFECTS ON PROCESS MATERIAL INTERACTION DURING MICRO MILLING

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The interrelationship between material microstructure and process parameters must be well understood in order to improve the machinability. In micro milling, the process parameters including depth of cut, feed and cutting edge radius are comparable to grain size of the material which significantly affects the mechanics of machining. This thesis investigates the contribution of microstructural characteristics including grain size, grain morphology and phase fractions/distributions of dual phase Ti6Al4V titanium alloy in micro scale milling. Various heat treatments were performed on the Ti6Al4V samples obtaining five different microstructures including fine equiaxed plus elongated, two size enlarged equiaxed, lamellar and martensitic microstructures. The influences of microstructures on built up-edge (BUE) formation, cutting forces, surface quality and burr formation were studied. It was observed that smaller grain size leads to larger BUE and burr formation and higher cutting forces. However, when feed is set properly it also yields better surface roughness. The crystallographic texture and microstructure of the machined surface of selected samples were investigated using electron backscatter diffraction (EBSD) analysis which revealed that at low feed rates can result in occurrence of dynamic recrystallization (DRX) on the microstructure of the machined surfaces. It was observed that up and down milling stages led to different crystallographic texture of the machined samples during micro scale milling. The findings of this study are important in terms of developing predictive modeling of machining based on material microstructure.

Keywords: Micro milling, Microstructure, Ti6Al4V titanium alloy, EBSD.
ÖZET

TI6AL4V MALZEMESİNİN MİKRO FREZELENMESİ ESNASINDA MİKRO YAPININ İŞLEME ÜZERINE OLAN ETKİLERİİNİN ARAŞTIRILMASI

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Anahtar sözcükler: Mikro frezeleme, Mikro yapılar, Ti6Al4V titanyum alemi.
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Chapter 1

Introduction

There exist various techniques in order to fabricate small products in micro scale. Mechanical micro machining processes such as micro milling, deliver variety of possibilities to manufacture small scale parts with high precision. In these operations, many factors including cutting tools, materials properties, process inputs etc. should be controlled and studied to attain a part with desired precision, properties and efficient energy consumption. Among these parameters, the material’s characteristics play an important role in every manufacturing process. Having gained an in-depth understating toward the history and physical/mechanical properties of the workpiece, one is able to predict and tune the outputs of the process in a desired manner [1, 2, 3].

Among the vast variety of engineering materials such as metals, polymers, ceramics and composites, metallic parts are highly considered thanks to the superior combination of strength, ductility, formability, etc. Titanium alloys have many applications in science and industry owing to their excellent properties including high strength, corrosion resistant and biocompatibility. However, these alloys are known as difficult-to-cut materials with low thermal conductivity. High extent of tool wear and manufacturing costs can also be mentioned as other problems of these alloys arising during machining processes [4, 5, 6].
Microstructural issue is one of the key factors in multi-scale machining processes. Due to heat generation and severe plastic deformation occurring during machining processes, a detailed observation of microstructural behavior of the machined workpiece is essential. Material microstructure in machining incorporates hardening, softening, phase transformation, grain refinement and recrystallization phenomena. Depending on the conditions of the employed machining process several of the mentioned issues might take place [7, 8]. Therefore, thermomechanical-microstructural approaches are required to avoid undeniable changes while machining to enhance the quality of the final products. In the literature, most of the machining-microstructure investigations are dedicated to macro scale machining processes either experimentally or computationally. Shivpuri et al. [9] studied the formation of chips as a result of microstructural changes in high speed machining of Ti6Al4V alloy. They reported that, when the cutting speed is low, the shapes of the chips are discontinuous, while by increasing cutting speed and reaching $\beta$-transus phase transformation temperature, chips become continuous and segmented. Wan et al. [10] analyzed the adiabatic shear bands (ASBs) formed in machining and mechanism of chip formation in a microstructural-based approach for a titanium alloy. They reported that, width of adiabatic shear bands increases by increasing cutting speed, feed rate and decreasing tool rake angle. They showed that, microstructural evolution in shear bands depend on cutting speed. Phase transformation from $\beta$ phase to martensitic microstructure in adiabatic shear band was also shown in their study. Campbell et al. [11] investigated microstructure changes during high speed macro machining of an aluminum alloy. They showed that, precipitate growth and grain recrystallization can take place in both adiabatic shear bands and produced chips. Shankar et al. [12] also reported severe grain refinement in the sample as a result of high strain in high speed machining. Pan et al. [13] studied material behavior under machining in various conditions by applying predictive models. They used Johnson-Mehl-Avrami-Kolmogorov (JMAK) to address grain growth and recrystallization during high speed orthogonal machining of Ti6Al4V. They reported the rise of beta phase fraction by increasing feed rate. Many studies also have reported the formation of white layer on the surface of machined alloys. For instance, Barry and Byrne [14] analyzed the white layer formed in machining of a steel. They
showed the dynamic recrystallization (DRX) phenomenon occurs in white layer. For titanium alloys, Che-Haron and Jawaid [15] reported the formation of white layer accompanied by severe grain refinement. They also demonstrated the effect of white layer and microstructure on the surface integrity of machined workpieces. Armendia et al. [16] compared the machinability of two different titanium alloys Ti6Al4V and Ti54M. These alloys have similar material properties, but Ti54M is known to have better machinability. The study’s results show that the differences in microstructures are responsible for better machinability of Ti54M. The size of BUE formed while machining Ti54M was observed to be smaller which contributes to better machinability, but the relationship between the microstructure and BUE formation was not investigated. Sun et al. [17] studied the effect of microstructure of Ti6Al4V alloy in high speed turning process. They presented the possibility that segmented chip formation would be higher for fully lamellar microstructure compared to bimodal microstructure. Abbasi et al. [18] also reported that the lamellar microstructure obtained after heat treating of titanium alloy Ti6Al4V yielded better machinability when polycrystalline diamond cutting tools were used. Deng et al. [19] reported that grain refinement occurs due to large shear strains during high speed machining, which can be utilized to produce ultrafine grained material. Cedergren et al. [20] analyzed machinability of three different microstructures of titanium alloy during orthogonal turning and reported that feed and work material microstructure directly affect chip formation. Nouari and Makich [21] reported the influence of different microstructures in titanium alloys on tool wear. They reported the presence of intense deformation zone beneath the machined surface and hence subsurface texture modification using SEM-EDX analysis. Pan et al. [22] showed that surface integrity as one of the most critical outputs of multi-scale cutting operations can be affected by material microstructure. They also reported that, residual stress, chip formation, cutting forces all depend on material condition.

As it was reviewed in the aforementioned studies, the interaction between material microstructure and process parameters can vary based on the condition of the experiments. However, it can be predicted that, owing to severe plastic deformation and thermomechanical effect in multi-scale machining operations,
one may expect the influence of microstructure and also microstructure induced changes. In addition to the direct control over process parameters including feed rate, spindle speed, tool condition etc., a detailed microstructural characterization needs to be performed to address the gap between material properties and machining topics.

In micro scale machining like micro milling, process settings are set such that feed, depth of cut and cutting edge radius of the micro end mill are in the same order of magnitude as the grain size of the material. Therefore, the anisotropic behavior of the multiphase materials considering their grain size, grain boundaries, and phase fractions must be studied. The microstructural features of a part are essential to be analyzed and controlled. Besides, how the material responses to a micro milling process and how is it affected in terms of microstructural and surface properties, would be some critical questions requiring profound studies. In addition, the selection of process parameters during machining of titanium alloy may also affect the surface texture and microstructure of the work material. The interplay between machining process parameters and the microstructure of polycrystalline dual phase materials such as titanium alloy Ti6Al4V requires detailed investigations. A better understanding of the relationship between material microstructure and its machinability will help develop predictive machining models that reduce experimental work performed during the process design phase.
Objectives

In this thesis, using a multidisciplinary approach consisting of manufacturing, metallurgy and materials science, a detailed investigation toward the interrelationship between micro milling outputs and material microstructure of Ti6Al4V titanium alloy has been carried out. The machinability of various Ti6Al4V microstructures are aimed to be analyzed. For this purpose, by utilizing heat treatment technique, various microstructures of Ti6Al4V have been attained and studied during micro milling experiments. The influence of micro milling process on microstructure and crystallographic texture of the material have been also analyzed in this study. In summary, the main objective of this study is to enhance the understanding toward material/process interaction in micro scale milling by considering microstructural features including grain size, grain morphology and phase fractions/distributions.

Organization of the thesis

This thesis is organized in such a way that, in chapter 2, the principles of titanium alloys, sample preparation, heat treatment routs, and determination of material properties are discussed. In chapter 3, micro milling experiments and various outputs of different machined samples including cutting forces, built-up edge formation, surface roughness and burr formation are analyzed in detail. In chapter 4, the results of microstructure and crystallographic texture analyses using electron backscatter diffraction (EBSD) techniques are studied. Finally in chapter 5, utilizing the results from EBSD, 3D microstructural construction is conducted by means of Dream3D package in order to be used for further simulation studies.
Figure 1.1: The flowchart of present thesis.
Chapter 2

Sample preparation, heat treatments and determination of material properties

2.1 Introduction

Titanium alloys are acknowledged for their widespread applications owing to their excellent properties such as high strength, corrosion resistance and biocompatibility among the engineering alloys. These alloys exhibit versatile types with specific properties depending on the utilized composition and production method. In addition to pure titanium, this alloy can be seen in the forms of near $\alpha$, $\alpha+\beta$, near $\beta$ and $\beta$ alloys. In these alloys, $\alpha$ phase exhibits hexagonal close packed (HCP) crystal structure, whereas $\beta$ phase exists with body centered cubic (BCC) crystal structure. By tuning the alloy with $\alpha$ or $\beta$ stabilizing elements, one can reach a preferred type of titanium alloy based on the targeted application. Aluminum, oxygen and carbon are most common $\alpha$ stabilizers, while molybdenum and vanadium are used as $\beta$ stabilizers within this alloy [23, 24, 25, 26].
Deformation mechanism in titanium alloys are mostly associated with slip and twinning due to their high stacking fault energy (SFE). Depending on mode of deformation and state of stress and strain, various anisotropic deformation can be noticed in specially α+β alloys. Since most of the fraction of α+β alloys is dedicated to α phase, the deformation is expected to be more affected by this phase. Due to asymmetric structure of HCP α phase, a complex deformation mechanism can take place within these alloys. Basal, prismatic and pyramidal slip systems or combination of them can be activated while deforming the sample. These slip systems for α phase are shown in Figure 2.1, {0001} for basal, {1010} for prismatic and {1011} for pyramidal planes. Normally, the [1120] slip direction accompanied by the mentioned slip planes form the deformation mechanism by slip in this alloy [26, 27, 28].

![Diagram of major slip planes for α titanium](image)

Figure 2.1: Major slip planes for α titanium, adopted from [28].
Ti6Al4V, is the most common used and studied $\alpha + \beta$ type titanium alloy in science and industry. Figure 2.2 illustrates the phase diagram of this alloy where point ‘a’ designates $\beta$-transus in about 980 °C [26].

By tailoring the thermomechanical and heat treatment paths, it is possible to obtain various microstructures out of Ti6Al4V. Equiaxed, bimodal, lamellar, and martensitic microstructures can be named as most common outputs of heat treatments of this alloy [29].

There are many studies in the literature reporting various material conditions and mechanical properties of Ti6Al4V. For instance, Lee and Welsch [30] reported the effect of microstructure on elastic behavior of Ti6Al4V. They showed that, Young’s modulus of this alloy is dependent on the existing phases and the applied heat treatment cycles. Venkatesh et al. [31] investigated the hardening behavior, strain rate sensitivity and fracture mechanisms of this alloy as a function of various heat treatments. They reported that yield strength and ultimate tensile strength and hardness of various heat treated samples were affected by the present phases including $\alpha$, $\beta$ and martensite. Stefansson et al. [32] investigated
the mechanism of globularization of Ti6Al4V and declared that formation and evolution of dislocation substructure are responsible for two stage globularization. Jovanović et al. [33] extensively studied the effect of annealing temperature and cooling rate on mechanical properties and microstructure of Ti6Al4V alloy. They concluded that, hardness values and tensile strength can be improved by increasing annealing temperature and cooling rate during heat treatment. While, the higher annealing temperature and cooling rate, results in the lower elongation. Filip et al. [34] also studied the mechanical properties of this alloy in various microstructural conditions. They reported that, due to geometrical factor of $\alpha$ phase, with increase of aging temperature and time, the fracture toughness decreases. It was also mentioned that, volume fraction of primary $\alpha$ phase can result in reduction of fracture toughness of Ti6Al4V. Ahmed et al. [35] analyzed the phase transformation behavior of this titanium alloy and reported the formation of martensitic structure and Widmanstätten phase. Elmer et al. [36] investigated the lattice structure behavior of Ti6Al4V by an in-situ approach. It was revealed that, intense differences were detected in the changes of the lattice parameters of $\alpha$ and $\beta$ phases during the transformation process. The observed alterations in lattice structures were ascribed to vanadium element and its positioning. They also showed the presence of residual stress caused by heat treatment. It is worth mentioning that, the extent of additional elements can affect the properties of Ti6Al4V in various deformation processes. For instance, Zong et al. [37] studied the influence of addition of hydrogen within the composition of the alloy. They claimed that, dislocation density, stacking fault energy and mechanical properties can be affected by small amount of hydrogen addition. Khan et al. [38] reported the effect of oxygen on microstructure and thermomechanical behavior of this alloy indicating that the composition of the utilized titanium alloy should be considered thoroughly.
2.2 Sample preparation

Samples were prepared with the dimensions of $20\text{(mm)} \times 20\text{(mm)} \times 5\text{(mm)}$ by considering the appropriate size for micro-scale milling process and materials characterization methods. Figure 2.3(a) illustrates the typical shape and dimensions of the fabricated sample. Based on the locations of the screw places provided in the Dynamometer stage, four holes were drilled within the sample in order to clamp the workpiece firmly on the Dynamometer. The composition of the as-received Ti6Al4V alloy is demonstrated in Figure 2.3(b) by EDX analysis, indicating the nominal composition of Ti6Al4V alloy.

![Figure 2.3: (a) Typical shape and dimensions of the utilized workpiece, (b) EDX spectra of the as-received Ti6Al4V alloy.](image)
2.3 Heat treatments

In order to obtain various microstructures of Ti6Al4V alloy, the heat treatments were performed using a high temperature furnace up to 1100 °C. For this purpose, initially the samples were cleaned by ethanol to avoid further contamination in heat treatment process. The typical phase diagram of Ti6Al4V alloy is provided in Figure 2.4 containing important heat treatment and phase transformation points. In this work, in addition to the as-received sample (S1), different heat treatment paths were manipulated as following. The detailed information of sample labels and applied heat treatment for each specimen are given in Table 2.1.

- Heating to 925 °C (point ‘c’ in Figure 2.4) for 4 and 24 hours (two samples) followed by furnace cooling.

- Heating to 1035 °C (point ‘a’ in Figure 2.4) for 30 minutes followed by furnace cooling.

- Heating to 1035 °C (point ‘a’ in Figure 2.4) for 30 minutes followed by water quenching.

![Figure 2.4: Phase diagram of Ti6Al4V containing heat treatment points.](image-url)
### 2.4 Metallography and microstructural characterization

At the beginning, the cross sections of the samples were chosen to perform metallography. In order to remove the damages caused by sectioning step, the specimens were ground using 80 to 2000 grinding papers. In every level of grinding scratches orientation was precisely noticed in order to be in the same direction.

Afterward, the polishing step was done using 6 to one micro meter diamond suspension on the nap cloth. The rotation speed of the polisher was defined as 250 rpm in order to accurately notice the polishing process. The polishing step was categorized into three steps. During the initial polishing stage 6 micro meter diamond suspension was used. In the intermediate and final stages, 3 and one micro meter diamond suspension were utilized respectively.

Finally, the samples were washed and prepared for etching stage. The well-known Kroll’s etchant (6 ml HNO3 + 2 ml HF + 92 ml distilled water) was manipulated to etch the surface of the sample. Generally, etching time varies depending on the composition of the alloy. Using trial and error of every 3 seconds intervals, 15 seconds etching time was found to be enough to reveal the anticipated microstructure.

Employing Keyence VKX-1000 digital microscope, optical micrographs of the specimens were taken. The image analysis of the captured optical micrographs were carried out thoroughly by means of MIP and ImageJ software [39].

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Sample label</th>
<th>Holding temperature (°C)</th>
<th>Holding time (h)</th>
<th>Cooling type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>S1</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Sample 2</td>
<td>S2</td>
<td>925</td>
<td>4</td>
<td>furnace</td>
</tr>
<tr>
<td>Sample 3</td>
<td>S3</td>
<td>1035</td>
<td>0.5</td>
<td>furnace</td>
</tr>
<tr>
<td>Sample 4</td>
<td>S4</td>
<td>1035</td>
<td>0.5</td>
<td>water</td>
</tr>
<tr>
<td>Sample 5</td>
<td>S5</td>
<td>925</td>
<td>24</td>
<td>furnace</td>
</tr>
</tbody>
</table>
goal of performing image analysis has been identifying the grain size, morphology and volume fraction of each phase within various samples in this research. It is worth mentioning that, the method applied in the image analysis step has been according to ASTM E-112-96 standard encompassing interception analysis using 50 horizontal and vertical lines in the grid matrix.

It should be noticed that, before performing image analysis and further micro milling tests on the heat treated samples, the $\alpha$ case layer created on the surface due to oxidation, was removed from the samples by grinding technique. This hard oxide layer is manifested in Figure 2.5. Alpha case is very hard and brittle and promotes the formation of microcracks which will reduce the material response to mechanical deformations. It is worth mentioning that, higher temperatures and heat treatment durations may result in increasing the thickness of this layer.

![Figure 2.5: Illustration of $\alpha$ case layer region.](image-url)
The optical micrograph together with phase fractions attained by image analysis of the as-received specimen are shown in Figure 2.6. Apparently, the revealed microstructure consists of equiaxed $\alpha$ plus elongated grains and $\beta$ phase in the grain boundaries of $\alpha$ phase. As illustrated in Figure 2.6(b), the fractions of $\alpha$ and $\beta$ phase were found 84% and 16% respectively.

Figure 2.6: (a) Optical micrographs of the as-received alloy with the analyzed image (b) the fractions of existing phases.

Figure 2.7 depicts the optical micrographs and phase fractions of the sample heat treated in 925°C (point c in Figure 2.4) holding for 4 hours followed by furnace cooling. This microstructure includes equiaxed $\alpha$ grains and $\beta$ phase distributed homogeneously. As shown in Figure 2.7(b), the fraction of $\alpha$ and $\beta$ phase were attained 78% and 22% respectively.
Figure 2.7: (a) Optical micrographs of the S2 sample with the analyzed image (b) the fractions of existing phases.

Figure 2.8 depicts the optical micrographs and phase fractions of the sample heat treated in 925°C (point c in Figure 2.4) holding for 24 hours followed by furnace cooling. This microstructure includes fully equiaxed α grains and β phase. As shown in Figure 2.8(b), the fraction of α and β phase were attained 74% and 26% respectively. This indicates the rise of β phase fraction in comparison with two previous samples.

The optical micrograph and image analysis results of the specimen heated to 1035°C (point a in Figure 2.4) holding for 30 minutes followed by furnace cooling, are provided in Figure 2.9. This microstructure is mainly composed of plate-like
Figure 2.8: (a) Optical micrographs of the S5 sample with the analyzed image (b) the fractions of existing phases.

lamellar $\alpha$ and $\beta$ phase in grain boundaries. It has been shown that, due to low cooling rate of the sample in this heat treatment path, $\alpha$-Widmanstätten phase also may form. As demonstrated in Figure 2.9(b), the fraction of $\alpha$ and $\beta$ phases in this sample were attained 72% and 28% respectively. Consequently, the fraction of $\beta$ phase has been increased in comparison with the as-received sample.
Figure 2.9: (a) Optical micrographs of the S3 sample with the analyzed image (b) the fractions of existing phases.
The average quantitative information of image analysis of various microstructures are summarized in Table 2.2. As it can be noticed, S1 exhibits lower $\alpha$ and $\beta$ grain sizes compared to S2 and S5. By performing heat treatment, both $\alpha$ and $\beta$ grain sizes increased substantially. For S5 case, this increase was very sensible with up to 38 $\mu$m grain size. Owing to lamellar shape of S3 microstructure, the average grain sizes in width and length of the phases are reported.

<table>
<thead>
<tr>
<th>Sample label</th>
<th>Microstructure type</th>
<th>$\alpha$ grain size ($\mu$m)</th>
<th>$\beta$ grain size ($\mu$m)</th>
<th>$\alpha$ phase fraction (%)</th>
<th>$\beta$ phase fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>Equiaxed+Elongated</td>
<td>4.9</td>
<td>1.6</td>
<td>84</td>
<td>16</td>
</tr>
<tr>
<td>S2</td>
<td>Equiaxed</td>
<td>9.6</td>
<td>2.3</td>
<td>78</td>
<td>22</td>
</tr>
<tr>
<td>S3</td>
<td>Lamellar</td>
<td>5 (width) 20 (length)</td>
<td>2.8 72</td>
<td>28</td>
<td>28</td>
</tr>
<tr>
<td>S5</td>
<td>Equiaxed</td>
<td>38</td>
<td>2.6</td>
<td>74</td>
<td>26</td>
</tr>
</tbody>
</table>

In order to better compare the outputs of heat heat treatments, The optical micrographs of microstructures of the all heat treated samples S1, S2, S3, S4 and S5 are demonstrated in Figure 2.10 respectively. As it was declared already, the microstructures of S1, S2 and S5 are consisted of mixture of $\alpha$ and $\beta$ phase with different grain sizes. Figure 2.10(c) depicts the lamellar microstructure and Figure 2.10(d) shows the S4 microstructure (the sample heat treated at 1035°C for 30 minutes and water quenched). The needle-like dark regions in this micrograph correspond to $\alpha'$-martensite phase which normally forms during fast cooling rates.
Figure 2.10: Optical micrographs of all samples microstructures, (a) S1, (b) S2, (c) S3, (d) S4, (e) S5.
In order to analyze the outputs of the heat treatment experiments illustrated in Figure 2.10, the metallurgical state of each microstructure is required to be discussed. The first heat treatment has been recrystallization annealing in the $\alpha+\beta$ region and holding for 4 hours and slow cooling. As shown in Figure 2.10(b), the $\alpha$ equiaxed grains were enlarged in comparison with the as-received sample due to acquiring sufficient annealing time (4 hours). The $\beta$ phase also increased in amount and size mainly in the boundaries of $\alpha$ phase as a result of annealing. The resulting microstructure is fairly coarse as was shown earlier by means of image analysis. As illustrated in Figure 2.10(c), the third microstructure is obtained by furnace cooling from above the $\beta$-transus resulting in lamellar microstructure. This microstructure sometimes refers to $\alpha$-Widmanstätten. The applied heat treatment path for this sample leads to nucleation and growth of $\alpha$ phase normally at the grain boundaries of $\beta$ phase. As it can be observed, $\alpha$ plate-like grains are fairly coarse surrounded by $\beta$ phase. The distinctive point about this microstructure is the presence of $\alpha$ grain boundary layer among the lamellar plate-like $\alpha$ phase. The next microstructure was attained by water quenching above the $\beta$-transus. Figure 2.10(d) depicts the resulting microstructure containing needle-like $\alpha'$-martensite. This phase is very critical in determination of mechanical properties and normally forms from prior beta phase within the microstructure. Many studies have reported formation of various martensite phases during thermomechanical processes of titanium alloys. The main in common point has been the high hardness of this phase. The microstructure of S5 shown in Figure 2.10(e) is also composed of enlarged equiaxed as a result of long time annealing (24 hours) in $\alpha+\beta$ region of phase diagram. In the next chapters, the principal differences among the attained microstructures are further analyzed and differences in their deformation behavior are discussed.
2.5 Micro hardness evaluation

Micro hardness evaluation is a useful way in order to determine the mechanical properties of materials and distinct phases. Vickers micro hardness testing (Zwick/Roell ZHVµ) was conducted on the samples with loads of 50 g and 500 g. Loads were applied for 10 seconds. Figure 2.11 shows the average results of Vickers micro hardness evaluations corresponding to two different loads. As it can be observed, for both applied loads, the maximum and minimum hardness values were found for S5 and S1 respectively. Also, the intermediate hardness values were obtained for S2 and S3 and S4. The considerably highest hardness of S4 is attributed to presence of hard martensite phase due to fast cooling above \( \beta \)-transus. Moreover, although the grain size of S1 was lower than that if S2, owing to higher fraction of \( \beta \) phase, the hardness value was achieved higher for the latter. For S5 case, due to long (24 hours) holding time in the furnace, enough time has been provided for hard elements to diffuse within the microstructure. In addition to the mentioned issue, increasing the fraction of \( \beta \) phase resulted in extreme increase of hardness in spite of having large grains for S5. The long holding time of S5 even made it much harder than S4 exhibiting martensitic microstructure. To elaborate, separate indentations with low amount of load equals to 10 g, were conducted on \( \alpha \) and \( \beta \) phases of S5 individually. The indent marks over these phases are depicted in Figure 2.12. As it can be noticed, the indent mark over \( \beta \) phase is smaller in diameter and less penetrated than that over \( \alpha \) phase, giving the hardness of 590 HV for \( \beta \) and HV Vickers for \( \alpha \) which are in good agreement with the previous literature [40, 41]. Consequently, the noticed hardness difference between S1 and S2 is mostly ascribed to difference in \( \beta \) fractions which were shown already in Table 2.2. On the other hand, S3 containing long lamellae of \( \alpha \) phase, and experiencing high temperature heat treatment up to 1035 °C, revealed lower value of hardness compared to enlarged equiaxed S2 and martensitic S4 samples.

In order to explain the observed difference in hardness values in \( \alpha \) (HCP) and \( \beta \) (BCC) phases, spot energy dispersive X-ray (EDX) analysis was carried out locally on each phase of S2 as presented in Figure 2.13 where \( \alpha \) and \( \beta \) phases are
indicated in the SEM image. The extent of aluminum and vanadium peaks are observed to be different for $\alpha$ and $\beta$ phases. EDX output of $\beta$ phase exposed a higher amount of vanadium and a lower amount of aluminum compared to that of $\alpha$ phase. Aluminum and vanadium are known as stabilizing alloy elements for $\alpha$ and $\beta$ phases, respectively [42]. These results demonstrate that the higher hardness value of $\beta$ phase is probably due to accumulation of vanadium and other elements. Therefore, in this dual phase titanium alloy, the detailed heat treat meant path consisting of time, temperature and cooling rate must be considered in order to explain the observed variations in hardness values.
Figure 2.12: Local micro hardness indents over α and β phases.

Figure 2.13: Spot EDX spectra over α and β phases.
Chapter 3

Micro milling experiments and process outputs

3.1 Introduction

One of the most frequently used material removal processes is milling operation. In this process, generally a rotating multi-tooth cutting tools (mills) are employed exhibiting relative motion (generated by a spindle system) with respect to the sample for removing the material [43]. The schematic representation of micro milling and its characteristics are illustrated in Figure 3.1. At the beginning of the process, one of the edges of the cutting tool starts removing the material. The first stage is called up milling where the uncut chip thickness starts from its minimum and reaches the maximum. This stage is begun with ploughing dominant region and end with shearing dominant region. Next stage is down milling, starting from maximum uncut chip thickness (feed value) to the end of the process. The x and y forces acting on the cutting tool during micro milling are also shown in Figure 3.1. There are some critical points which make micro milling somewhat different than macro scale milling in terms of process uncertainties. In micro scale milling, feed per tooth might be comparable to cutting tool edge radius. The run-out of the spindle and the utilized cutting tool may become larger than magnitude of
feed per tooth. Moreover, the grain size of the workpiece is generally comparable to depth of cut and feed. All the mentioned issues, give rise to difficulties in controlling the micro milling process [44, 45].

Figure 3.1: Schematic representation of micro milling process, adopted from [43].

As it was mentioned, in micro milling, process conditions are set such that feed, depth of cut and cutting edge radius of the micro end mill are in the same order of magnitude as the grain size of the material. Furthermore, due to high stress and strain values experienced by the sample, microstructure of the utilized material may undergo some damages or alterations. Therefore, the anisotropic response of the multiphase alloys regarding their grain size, grain boundaries and phase distributions need to be investigated [46]. The effect of the work material’s microstructure on its machinability at macro/micro scales has been
investigated in literature. The number of studies investigating the interaction between microstructure and micro scale machining are limited in the literature compared to macro scale machining. Attanasio et al. [47] conducted a detailed study on micro milling of titanium alloys with different microstructures. They reported that fully lamellar microstructure resulted in better tool life. Vogler et al. [3] studied the microstructure level force prediction in various types of grain size of a steel alloy. They reported the effect of initial grain size of the material on the nature of the out coming forces. Wu et al. [48] investigated the effect of grain size and cutting edge radius in micro cutting a titanium alloy. It was shown that smaller grain size yielded higher cutting forces and specific cutting energy. Komatsu et al. [49] showed that the smaller grain size of the material reduces burr formation. Imran et al. [50] utilized TEM and EBSD for characterizing the micro drilled nickel superalloy and reported the formation of nanocrystalline grains. Smaga et al. [51] used EBSD to investigate the microstructural changes in TRIP steel parts due to micro milling using. Lawson et al. [52] analyzed the effect of anisotropy of a single crystal aluminum in orthogonal micro machining, and reported the effect of anisotropic microstructure on the process outputs. Yet the interplay between microstructure and process outputs of micro scale milling is not completely understood. Therefore in the present thesis, in the following sections, a more comprehensive investigation regarding the influence of material microstructural features such as grain size, grain morphology and phase types on micro milling process are discussed.

### 3.2 Process setup and parameters

The setup of micro milling experiments is shown in Figure 3.2. Experiments were performed on a micro machining center (Mikrotools DT110). The initial process parameters for micro milling tests were kept the same for machining each sample, and they were set as follows: 28000 rpm spindle speed (35 m/min cutting speed), 30 µm depth of cut, 0.5, 1 and 1.5 µm/rev feed per tooth (28, 52 and 84 mm/min feed rates respectively), 0.4 mm tool diameter with two teeth (NS Tools MSE 0.4). Slots of 10 mm length were machined. A new micro end mill was used.
in each test. Kistler mini dynamometer (Type 9256) with its charge amplifier was used to measure cutting forces during micro milling experiments. A new set of experiments were performed for investigating the influence of feed rate on the microstructure and surface quality of the samples. Therefore, 0.5 and 3 \( \mu m/\text{rev} \) feed per tooth corresponding to 28 mm/min and 168 mm/min feed rates values were applied respectively. The mentioned parameters were selected to first satisfy the conditions of micro scale milling and second to minimize the uncertainties as much as possible. Final set of experiments were performed on all the samples with equiaxed microstructures exhibiting various grain sizes. The next sections are assigned to analyze the results of the employed micro milling conditions.

![Figure 3.2: The setup of micro milling experiments.](image)
3.3 Built-up edge (BUE) formation

Built-up edge (BUE) can significantly affect the surface quality and tool life during micro scale machining. It usually forms in specific cutting parameters by adhering to cutting tool edge in the tool–chip interface at the rake face. This phenomenon has been extensively studied in the literature on various machined materials [53, 54, 55]. However, the influence of various Ti6Al4V microstructures on the formation of BUE has not been addressed in detail. Moreover, the condition of the cutting tools and BUEs are the important factors contributing in obtaining different micro milling forces. It is important to notice how the microstructure affects the machinability of the work material. Figure 3.3 represents the scanning electron microscopy (SEM) images of the edges of four utilized micro milling tools after machining. Here, tools are labeled based on the corresponding machined sample label. For instance, T1 refers to the tool used for micro milling of S1. Images in Figure 3.3, indicate the formation of built-up edge (BUE) accumulated on the rake face of all the utilized micro milling tools. As it can be noticed, the created BUEs are essentially different in shape and amount. These images also denote that, at least for one of the edges of the employed cutting tools except T4, some damages on the tool coating were observed. The presence of some micro chips attached to BUEs can be observed in the images. The quantified areas of BUEs for each tool including both edges is presented in Figure 3.4 using image analysis.
Figure 3.3: SEM images of cutting tool edges (a) T1, (b) T2, (c) T3 and (d) T4 (left and right images are associated with edge 1 and edge 2 respectively).
Based on the results presented in Figure 3.4, S1 with lowest hardness and fine grain size resulted in highest amount of BUE on its corresponding tool (T1). As it has been recently found by Oliaei and Karpat [56], the presence of BUE can increase the cutting force. Hence, it can be concluded that, since the highest amount of BUE among all of the machined samples was found for S1, this phenomenon contributed in drastic increase of the resultant force of S1 relative to the other samples (next section in Figure 3.7). S4 with high hardness revealed less amount of BUE than S1 and S3. This can be also responsible in observation of the lowest force achieved for this sample (next section in Figure 3.7). S2 and S3 with higher hardness and $\beta$ fraction, exhibited lower area of BUE. Also, S2 with large grain size and intermediate hardness value exposed the lowest amount of BUE. Furthermore, the morphology of the BUEs differs for each tool and sample. S1, S2 and S3 resulted in layer by layer BUE, whereas S4 revealed kind of compacted BUE on its corresponding micro milling tool edge. Thus, to analyze the variations of cutting forces, the formation of BUE on the edges of the tools should also be taken into consideration. The discussed results revealed that, decreasing the grain size and enriching $\beta$ phase fraction in dual phase Ti6Al4V microstructure, may decrease the amount of BUE formation in micro milling experiments.
The corresponding SEM images of cutting tools used in separate micro milling of different equiaxed samples (S1, S2 and S5) are also illustrated in Figure 3.5. As it can be noticed, again S1 exhibited large amount of BUE on the tool edge. The presence of some serrated chips are interesting on this tool. The important point about these images is the observed damages on the tool used for S5 machining as shown in Figure 3.5(c). This tool damage increases the area of the cutting tool and resulted in extreme increase in cutting forces which will be presented in next section.
Figure 3.5: SEM images of the cutting tools (a) T1, (b) T2 and (c) T5.
3.4 Micro milling forces

The captured forces versus time of micro milled samples at 1.5 fpt feed in the initial set of experiments are depicted in Figure 3.6. The measured average peak to valley forces in x, y and z directions are also provided in Figure 3.7 after performing micro milling experiments on the samples.

Figure 3.6: Graphs of forces vs time for miro milled (a) S1, (b) S2, (c) S3 and (d) S4.

As it can be observed, the measured forces for all the machined samples, were obtained higher in x direction compared to y and z directions. The obtained variation in micro milling forces in this study, can be attributed to two main reasons. The first cause is the mechanical properties and microstructures of the
machined samples, and the second is presence of BUE which was discussed in the previous section. By comparing hardness values given in Figure 2.11, and the measured forces in Figure 3.7, one can notice that, the forces and hardness values exhibit almost inverse relationship. The lowest force was acquired for S4 which exhibited the highest hardness value. Therefore, higher hardness value does not necessarily result in higher milling force in micro scale cutting. To elaborate this issue, one should notice that, deformation behavior of a metallic sample is not solely affected by hardness value or yield strength as demonstrated by Ankem et al. [40]. As shown previously in Figure 2.10 and Table 2.2, the microstructure of S1 consisted of fine grain equiaxed and elongated grains, whereas S2 sample exhibited enlarged equiaxed microstructure (S2 grain size is almost two times of S1). Consequently, due to the finer grain size in S1 (both $\alpha$ and $\beta$ grains), the amount of obstacles for dislocation motions including grain boundaries were much more than those of S2 during severe plastic deformation occurring in the interface of the tool tip and the material while micro milling. In fact, as it has

Figure 3.7: Average peak to valley forces.
been shown in Sergueeva et al. [57] and Dieter and Bacon [58] the ductility and toughness of the fine grain size microstructure is normally higher than those of having large grain size microstructure resulting in more strain hardening. Moreover, as it has been demonstrated by Jun et al. [59], the higher fraction of $\beta$ phase reduces the ductility and toughness of Ti6Al4V alloy in small scale deformations. In addition, Jiang [60] indicated the annealing treatment of Ti6Al4V under high temperatures can increase the hardness of the sample, however, it reduces the ductility. Therefore, the differences in grain size, grain morphology and phase fraction affect the mechanical properties of titanium alloy, especially ductility and toughness, which can be the main factors responsible for the observed force variations in micro milling experiments. Thus, since S2 contained more fraction of $\beta$ phase in comparison with S1, its lower ductility and toughness led to lower micro milling resultant force. Considering other samples, the presence of martensitic microstructure in S4 provides a high hardness and yield strength, while it deteriorates the ductility and toughness of this sample as reported by Fan et al. [61]. The low force for S4 compared to other machined samples, is mainly due to the low tendency of this sample for enduring ductile plastic deformation, exhibiting brittle-like behavior during micro machining process. Also notice that, S3 containing lamellar microstructure and higher $\beta$ phase fraction compared to S1 and S2 (provided in Table 2.2), exhibited lower toughness and ductility resulting in lower force. Therefore, the difference in grain size, grain morphology and phase fraction affects both ductility and toughness of the samples which can be the main factors responsible for the observed force variations in micro milling experiments. Another important consideration on the machining forces was the condition of the cutting tool edges, which was discussed in the previous section.

The next set of experiment were conducted to investigate the effect of different feeds and microstructures. The average peak to valley forces are measured in each condition and given in Figure 3.8. Figure 3.9 represents the force versus time graphs for S1, S2, S3 and S4 micro milled samples with 0.5 fpt, 1 fpt and 1.5 fpt feed values. Here $z$ forces are ignored in the measurements due to their small magnitudes.
Figure 3.8: Average peak to valley forces for various feeds and samples.
Figure 3.9: Forces versus time with various feed rates for different samples (a) S1, (b) S2, (c) S3 and (d) S4.
Figure 3.8 and Figure 3.9 imply that, similar to previous series of experiments, S1 exhibited higher cutting forces both in x and y directions in comparison with other samples. A important observation is the rise of cutting forces by increasing feed rates for all the machined samples. The similar behavior was noticed in reduction of cutting forces from S1 to S4. It should be mentioned that, the amount of tool run-out somewhat affected the mentioned results. Tool run-out was observed to be higher for S1 compared to other workpieces. It can be mainly attributed to formation of large amount of Built-up edge which will be discussed in the next section.

In order to analyze the effect of various feed rates on the microstructure, forces and surface quality outputs, a new set of experiments were conducted on the same material (S1) with similar depth of cut and spindle speed. Resulting average peak to valley forces are given in Figure 3.10. The corresponding captured forces versus time of micro milling are depicted for three feed values of 0.5 fpt, 1.5 fpt and 3 fpt in Figure 3.11. As it can be noticed, by increasing feed values, all the forces in x, y and z directions are increased.

Figure 3.10: Average peak to valley forces for different feed values.
Figure 3.11: Forces versus time with feeds of (a) 0.5 fpt, (b) 1.5 fpt, and (c) 3 fpt.
3.5 Surface roughness

Surface quality of a part is probably the most important output of a machining process. The surface roughness analysis of the machined surfaces after micro milling experiments is discussed in this section. Three-dimensional topographies of the machined surfaces obtained using a laser scanning microscope (Keyence VK-X100) are given in Figure 3.12. The recorded images were analyzed based on ISO 25178 using MountainsMap ver7 software.

Figure 3.12: 3D surface topographies of (a) S1, (b) S2, (c) S3, (d) S4.
Areal surface roughness parameters of the machined surfaces are depicted in Figure 3.13, where $S_q$ represents root mean square and $S_a$ represents average areal surface roughness values. The results imply that surface roughness values are close to each other, but the surface roughness of $S_2$ is slightly greater than $S_1$. Considering that the same machining parameters were used in the experiments, it is expected to have similar surface roughness values. The influence of larger BUE on $T_1$ does not seem to affect the surface compared to smaller BUE on $T_2$. Long term machining experiments would reveal more reliable information on the surface roughness as shown in [62]. Among the machined surfaces, $S_4$ specimen ranks second in term of surface quality exhibiting smaller surface roughness. $S_2$ and $S_3$ are found to be very similar in surface roughness value. Therefore, $S_1$ with fine equiaxed microstructure and $S_4$ with martensitic microstructure delivered the best surface qualities compared to other samples.

![Figure 3.13: Areal surface roughness parameters of the machined surfaces.](image)
The 3D surface topographies of machined S1 at feed values of 0.5 fpt, 1.5 fpt and 3 fpt are demonstrated in Figure 3.14. The effect of feed rate on resulting surface should be investigated. As this figure shows, at low feed (0.5 fpt), the surface exhibits some small spikes deteriorating the surface quality. By increasing feed to 1.5 fpt, the surface quality become better, yet some tiny sharp spikes are observed in this surface which might be formed due to BUE. Finally, at 3 fpt, the surface quality is extremely improved. Thus, with the same microstructural and experimental parameters, one can attain better surfaces by increasing feed rates. However, higher feeds mean needing more cost and cutting energy.

![Figure 3.14: Surface of S1 machined at (a) 0.5 fpt, (b) 1.5 fpt and (c) 3 fpt.](image-url)
3.6 Burr formation

One of the undesirable outputs of a machining process is burr formation. This section is assigned to study the formation of burrs adjacent to the borders of the micro milled channels. The resulting 3D representation of burrs for each machined sample with different microstructures are provided in Figure 3.15. The average of burr height associated with each sample is given in Figure 3.16.

![Figure 3.15: 3D representation of burrs for (a) S1, (b) S2, (c) S3 and (d) S4.](image-url)
As it can be observed from the given figures, S1 and S4 exhibited highest and lowest amount of burrs among the machined samples respectively. The main reason behind these outcomes would be the differences in corresponding mechanical properties and hardness for the studied workpieces. S1 containing fine equiaxed microstructure and low hardness exposed high amount of burr. In fact, based on the attain results, the more ductile the material, the more amount of burrs would be expected. S4 with lowest ductility but highest hardness showed lowest amount of burrs. S3 also revealed low amount of burr very similar to S4. Thus, various microstructure conditions can also affect burr formation during micro milling process.

![Figure 3.16: Mean burr heights of all samples.](image-url)

Figure 3.16: Mean burr heights of all samples.
3.7 Discussion

The employed micro milling conditions in this thesis are included in the size effect region of micro milling. As it has been studied by Rezaei et al. [63], milling experiments with feed per tooth values up to approximately 3 fpt, are regarded in the region of micro milling size effect. It means, at feeds lower than 3 fpt, a drastic increase of specific cutting force (SCF) has been observed. Therefore, applying various types of Ti6Al4V microstructures and working in the size effect region is sufficiently challenging which requires further investigations. Based on the attained results of this study, a graphical representation of micro milling outputs and microstructure factor as an input, are illustrated in Figure 3.17. It was shown that, microstructure of the workpiece directly/indirectly affects micro milling outputs including cutting forces, built-up edge, surface roughness and burr formation. All the mentioned parameters in Figure 3.17 along with tool run-out, tool deflection etc. increase the uncertainties in controlling micro scale milling process. To investigate the specific additional effect of grain size, some new series of micro milling tests were performed on all equiaxed samples with different grain sizes (S1, S2 and S5) with 28000 rpm spindle speed, 30 µm depth of cut, 1.5 µm/rev feed per tooth (which is in the size effect region). The average peak to valley forces are provided in Figure 3.18. Corresponding force versus time of micro milling for these experiments are shown in Figure 3.19. In the previous experiments, it was shown that by enlarging grain size within the microstructure, one can attain lower cutting forces. However, the obtained forces associated with S1 and S2 samples in the new experiment are found to be slightly lower than those of previous experiments. When it comes to S5 with coarse grain microstructure, an increase in force magnitudes are revealed. This observation is probably ascribed to high hardness of this sample which result in some damages in the cutting tool edge which was designated in Figure 3.5. Thus, the condition of cutting tool is extremely significant in analysis of cutting force variations. In addition, the microstructural anisotropy and inhomogeneity of the different utilized samples might have affected these forces. Further studies in the same experimental situation may be required to conclude the size effect and microstructure relationship in detail.
Figure 3.17: Summarized results of the interplay between microstructure and micro milling outputs.

Figure 3.18: Average peak to valley forces for S1, S2 and S5.
Figure 3.19: Forces versus time for machined (a) S1, (b) S2, and (c) S5.

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Chapter 4

Electron Backscatter Diffraction (EBSD) analysis

4.1 EBSD procedure

Quantitative microstructural and crystallographic texture analyses play a critical role in determination of mechanical properties and deformation behavior of an alloy. Electron backscatter diffraction (EBSD) is a useful technique to examine the crystallographic behavior of the group of grains within a material that has undergone a manufacturing process. This method is widely used in the literature to study the microstructural characteristics within various metallic samples in different manufacturing processes [64, 65, 66]. This technique can generate information about the grain structure (size and distribution), grain boundary characteristics, crystallographic texture, and phase distribution. Guo et al. [67] analyzed surface deformation and microstructure in macro orthogonal cutting of brass and copper using EBSD analysis. They discussed formation of various microstructural changes including cellular and equiaxed grains as a result of different cutting speeds and shear strains. Velásquez et al. [68] investigated the deformation layers created due to high speed machining in Ti6Al4V using EBSD. Also using EBSD, Acharyya et al. [69] reported grain fragmentation and work
hardened layer as a result of machining of stainless steel. Crawforth et al. [70] reported the microstructure damages caused by precision turning of a titanium alloy. They used EBSD analysis to show that the depth of subsurface deformation varies according to cutting speed. M’Saoubi et al. [71] provided a comparison of cutting tool types and the subsequent influence on the surface integrity of machined Inconel 718. They indicated the presence of recrystallized grains near the surfaces of the machined parts due to high speed turning experiments. Using EBSD tool, Telrandhe et al. [72] showed that machining forces were considerably decreased after annealing and sub-surface residual stresses were revealed to be reduced. Another important point in their study was deformation twinning was observed on samples with high grain size which were annealed at a higher temperature. They concluded that, initial strain free grains and deformation twinning while machining lessens the cutting force at very high cutting speed.

To the best of the author’s knowledge, in the literature no research work has been conducted to address the texture and microstructural evolution after performing micro scale milling on Ti6Al4V. Thus, in this thesis EBSD technique and orientation imaging microscopy (OIM) were used to examine the micro milling induced microstructure alterations on the machined surface of S1 and S2 specimens. EBSD also permits for exact determination of grain size and phase fractions/distributions as well as the crystallographic texture behavior of the samples. EBSD analysis was conducted both on unmachined and machined surfaces of the S1 and S2 samples. Both up milling and down milling directions corresponding to the same uncut chip thickness were considered. In order to properly prepare the surface of the samples for EBSD investigation, they were first ground and polished mechanically. Afterward, the surfaces were prepared by electropolishing method using a chemical solution containing 60% methanol + 34% butoxyethanol + 6% perchloric acid with voltage of 60 V holding for 15 seconds at room temperature. In EBSD analysis, 25 kV accelerating voltage, 10 mm working distance and 0.12 μm step size were applied. The dynamic focus mode of SEM was utilized. The electropolishing setup and solution are shown in Figure 4.1. The electropolished as-received sample on EBSD sample holder is depicted in Figure 4.2(b). It must be noticed that, after performing electropolishing, the sample should be washed
by ethanol. Finally, the chamber of SEM/EBSD consisting of EBSD sample holder, camera, electron gun and the samples are given in Figure 4.2(a).

Figure 4.1: (a) Electropolishing setup, (b) Electropolishing solution.
4.2 EBSD results

Initial EBSD analysis was conducted on the unmachined surface of sample S1. Figure 4.3 illustrates the micro milling process along with a summary of EBSD results obtained from each region sized $50\mu m \times 50\mu m$. The orientation color maps, grain size distribution, and misorientation angle variation are demonstrated in Figure 4.4. The results in Figure 4.4(a) imply that, the unmachined S1 contained fine equiaxed grains accompanied by elongated grains toward the rolling direction (RD) of the sample. The fraction of $\alpha$ and $\beta$ phases were determined as 97.3%
and 2.7%, respectively. These results, which are calculated from a smaller area, differ from those calculated with images analysis in Table 2.2. EBSD results for areas corresponding to down milling and up milling direction of the machined S1 are given in Figure 4.5 and Figure 4.6, respectively.

The orientation and shapes of both $\alpha$ and $\beta$ grains were affected by micro milling experiments. Figure 4.4(s), Figure 4.5(a), and Figure 4.6(a) reveal that after performing micro milling, the $\alpha$ grains tended to orient and expand toward transverse direction (TD) while they were mostly aligned to rolling direction (RD) in unmachined S1. Furthermore, $\beta$ grains became smaller in size and got more
Figure 4.4: EBSD results of the unmachined S1; (a) orientation color maps, (b) grain boundaries map, (c) misorientation angle variation, (d) corresponding grain size distribution.

fragmented and scattered, giving lower fraction within the microstructure as a result of micro milling. The grain size distributions are presented in Figure 4.4(d), Figure 4.5(d), and Figure 4.6(d). The 8.1 $\mu m$ average grain size of unmachined S1 declined to 7.5 $\mu m$ and 7.7 $\mu m$ for down milled and up milled regions, respectively. The elongated grains observed on the unmachined surface are slightly reduced in size and assume more equiaxed shapes after micro milling for both down milled and up milled regions. Comparing Figure 4.4(c), Figure 4.5(c), and Figure 4.6(c) present an increase in high angle grain boundaries (HAGBs) which are considered boundary angles between $15^\circ$ and $180^\circ$ [73]. Misorientation angles were increased after performing micro milling. The percentage of HAGBs on unmachined S1 increased from 16% to 31.9% and 22.9% for down milled and up milled regions, respectively. The down milling region seems to be more affected by the micro milling compared to the up milling area.
Another observation in the EBSD results is the crystallographic texture of the grains. Considering inverse pole figures of unmachined (Figure 4.7(a)), down milled (Figure 4.7(b)) and up milled (Figure 4.7(c)) regions, the grains textured to a favorable orientation about 25° to [0001] direction designated by a red circle. On the other hand, for the up milling region, the resulting texture approaching mostly to [2110] orientation and distributed in the intermediate directions between [0001] and [2110]. Furthermore, inverse pole figure associated with the down milling region shown in Figure 4.7(b) is highly analogous to that attained for the forged (compressed) titanium parts reported by Kocks et al. [74], which implies that the material experienced higher compression in the down milling region compared to the up milling region. Based on the obtained inverse pole figures, the dominant deformation mechanism for the down milled area was mainly associated with activated basal slip systems of HCP titanium. However, for the up milling region,
it was the combination of basal and prismatic slip systems as shown in Bridier et al. [27]. Based on the obtained OIM images, no deformation by twinning was observed. According to Dillamore and Roberts [75], deformation by twinning is highly dependent on existing phases and mode of deformation to satisfy the condition of twin formation. Tensile rather than compressive stresses are required to form twins in HCP metals. In addition, for Ti6Al4V the slip mechanism mostly takes place rather than twinning.

EBSD results of the unmachined surface of S2, down milling, and up milling regions are presented in Figure 4.8, Figure 4.9 and Figure 4.10, respectively. For the unmachined surface of S2, a larger area (60µm × 60µm) was selected to observe more number of grains before conducting micro milling experiments.
As Figure 4.8(a), Figure 4.9(a) and Figure 4.10(a) depict, the fraction of $\beta$ phase obtained 2.8%, 4.4% and 2.3% which are generally higher than values found for S1. In contrast to S1, the $\beta$ grains detected were larger and less scattered in the machined sample S2. This result also supports the role of smaller $\beta$ grains which resulted in higher cutting forces for S1. $\beta$ grains mostly oriented to the favorable $[001]$ direction for the down milled area, and intermediate direction between $[111]$ and $[101]$ for up milled region designated by red and blue colors by unit triangle in Figure 4.9(a) and Figure 4.10(a), respectively. In addition, the average grain size of 18.8 $\mu m$ of unmachined S2, changed to 16.8 $\mu m$ and 18.4 $\mu m$ for down milling and up milling regions, respectively. Comparing Figure 4.9(c) and Figure 4.10(c), one can notice that for both cases, the low angle grain boundaries (LAGBs), i.e. the boundary angles between $1^\circ$ and $15^\circ$, were dominant. While the fraction...
of HAGBs were observed to be more for the down milled area, similar to the obtained results associated with machined S1. Based on the inverse pole figure shown in Figure 4.11, the texture of the grains acquired for down milling region of S2 (Figure 4.11(b)), is more focused in a specific orientation and is highly analogous to the textures on S1, indicating that deformation modes had the same influence during down milling of both the studied samples. Moreover, similar to S1, for the up milling area of S2 (Figure 4.11(c)), a more diverse texture was also observed. The results show that S1 experienced more microstructural changes than S2 during micro scale milling.

Based on the EBSD results obtained for S1, after performing micro milling experiments, the evidence denotes the highly possible occurrence of dynamic recrystallization (DRX) on the microstructure of machined surfaces. S1 was the
Figure 4.9: EBSD results of the down milled S2; (a) orientation color maps, (b) grain boundaries map, (c) misorientation angle variation, (d) corresponding grain size distribution.

as-received material containing work hardened microstructure. Mamedov and Lazoglu [76] and Thepsonthi and Özel [77] reported that localized temperatures can rise up to 700 °C at the tool-workpiece interface during micro milling of Ti6Al4V. Considering the low thermal conductivity of the titanium alloy, temperatures at the cutting zone may reach 0.1-0.5Tm (Tm is the melting temperature), which is the temperature interval for occurrence of DRX (Estrin and Vinogradov [78]). The temperature rise accompanied by a relatively high strain rate during micro milling may increase dislocation density and formation of subsequent cellular structure within the microstructure in recovery stage. This stage is followed by dynamic recrystallization (DRX) phenomenon resulting in grain refinement within the microstructure (Humphreys and Hatherly [73]). Such small new grains are designated in Figure 4.5(a) and Figure 4.6(b). Fragmentation of elongated grains accompanied by reduction of average grain size after micro
milling of S1, the procedure of grain refinements by DRX seems to have occurred. Further supporting evidence for this comes in the rise of HAGBs fraction on micro milled samples. In S2, a slight increase in HAGBs fraction was observed for down milling stage; however, no such grain refinement appeared in this sample. HAGBs were mostly revealed for $\beta$ grains after micro milling of S2. Therefore, the applied micro milling condition in this study produced no detectable dynamic recrystallization (DRX) within the machined surface of S2. This might be attributed to the large initial grain size and high fraction of $\beta$ as the second phase. Also, in case of S1, the microstructure was work hardened as-received and also consisted of elongated grains which became fragmented and refined after micro milling. Whereas in case of S2, the microstructure of the sample was fully annealed equiaxed. Based on the work of Huang and Logé [79], larger initial grain size leads to reduction of recrystallization kinetics, which may explain the lack of
not observed DRX in S2. It also should be mentioned that, the obtained results for machined S1 microstructure are more analogous to occurrence of continuous dynamic recrystallization (CDRX) rather than discontinuous dynamic recrystallization (DDRX) as shown by Humphreys and Hatherly [73]. Normally CDRX occurs at high stacking fault energy materials like titanium alloys and also it can occur in relatively low temperatures.
The previous EBSD analysis was associated with 1.5 fpt feed value. In addition, the surfaces of the machined S1 micro milled with 0.5 fpt and 3 fpt are also considered to be analyzed for possible microstructural evolution. The EBSD results for S1 micro milled with 0.5 fpt and 3 fpt are shown in Figure 4.12 and Figure 4.13 respectively.

Figure 4.12: EBSD results of the machined S1 with 0.5 fpt feed; (a) orientation color maps, (b) grain boundaries map, (c) misorientation angle variation, (d) corresponding grain size distribution.

By comparing these figures, it can be noticed that, by applying 0.5 fpt feed value, the elongated grains are still present and locate along the rolling direction (RD). Also no such increase in fraction of HAGBs are seen. The 8.1 µm initial average grain size of S1 before machining, reached to 7.9 for this feed value which are quite close. Whereas, for 3 fpt case, generation of small equiaxed grains within the microstructure are conspicuously observed. The fraction of HAGBs are also increased after micro milling of S1. The grain size after micro milling in this feed values declined to 7.2. Similar to the previous S1 machined sample with 1.5 fpt, the observed evidences for 3 fpt machined S1 also indicates occurrence of dynamic recrystallization (DRX). Therefore, it can be concluded that,
Figure 4.13: EBSD results of the machined S1 with 3 fpt feed; (a) orientation color maps, (b) grain boundaries map, (c) misorientation angle variation, (d) corresponding grain size distribution.

at very small feed rates like 0.5 fpt value, no such microstructural evolution was revealed. While by increasing feed to 1.5 fpt and 3 fpt, the generated heat and strain rate may have caused the sample to experience dynamic recrystallization. These results manifest that, micro scale milling process parameters are influential in occurrence of microstructural changes in term of DRX. As it has been studied by Landau, Rittel et al. [80, 81], the contribution of prior work hardening and stored energy in the sample is considerably important for DRX to occur. They have shown that, even in low temperatures the microstructure subjected to deformations may result in formation of new recrystallized grains in adiabatic shear bands which also contributed to ductile failure of the titanium alloys. It should be mentioned that, Ti6Al4V alloy exhibits various states of microstructures depending on the work material history. Thus, the focus of this thesis has been dedicated to equiaxed and elongated grain types with regular phase distributions within the microstructure of Ti6Al4V. Further investigations are required to study microstructural evolutions within other microstructure types of Ti6Al4V.
Chapter 5

3D construction of microstructure using Dream3D and EBSD results

Modeling of any manufacturing process can be very useful in order to first understand the process/material interaction and also to save time, cost and energy instead of performing lots of experimental works. In this thesis, a foundation was laid to correlate the microstructural features (grain size and phase fraction/distribution) and micro milling outputs of Ti6Al4V titanium alloy. In addition, the influence of micro milling process on the microstructure and crystallographic texture of the machined samples was thoroughly investigated using EBSD analysis. These findings can be implemented in computer modeling tools to advance the understanding and prediction of micro scale machining processes.

In the literature, a considerable amount of studies have examined the influence of microstructure in predictive modeling of machining of titanium alloys using finite element models. Zhang et al. [82] developed a microstructure-dependent flow stress model for two-phase titanium alloys. The material constitutive model captures the phase transformation and was integrated into finite element software to simulate chip serration in high speed machining of titanium alloy. They
noted the importance of $\beta$ phase in process outputs. Using the finite element method, Arısoy and Özel [83] investigated the microstructure alterations during face turning of Ti6Al4V titanium alloy, and they showed that recrystallization occurred within the microstructure due to temperature rise and the imposed high strain. Recently, Pan and Liang [22] also developed a microstructural framework to model process and material parameters in orthogonal cutting. They predicted dynamic recrystallization (DRX) via their proposed model for Ti6Al4V alloy.

Among many useful softwares, Dream3D package delivers an extensive possibility to digitally construct 3D microstructures by using results from EBSD technique. In general, DREAM3D is an open-source software package providing a high-level digital environment to create, segment, analyze and represent digital microstructure outputs. The main objective of Dream3D is to allow the interchange of microstructure representation to a digital source with user-friendly software tools. By implementing various filters in this environment, one can construct a preferred type of microstructures and use the output for further simulation purposes [84]. There are many research works utilizing this package regarding the analysis of microstructural and deformation processes on metallic samples [85, 86, 87]. As titanium alloys are concerned, in an interesting study, Ozturk et al. [86] used Dream3D to generate statistical microstructure of dual phase Ti6Al4V in order to study micromechanical behavior of the alloy. They reported that, the results are more sensitive to $\alpha$ phase fraction rather than $\beta$ phase. In addition, they included the effect of grain morphology, loading direction and inertial grain orientations in their study.

Hence, this package can be very useful to convert real data microstructure to digital representation. In this study, using two various approaches, microstructure construction was perform using Dream3D. The first approach has been, the statistical method by which the data of EBSD are implemented by the gained statistics. The second approach has been employing the raw data from EBSD and directly constructing the microstructure to obtain more realistic microstructure representation. Using this package, one can obtain various outputs including ABAQUS finite element input file, orientation distribution function (ODF) maps, inverse pole figures (IPF) and can be visualized by ParaView software.
5.1 Statistical approach

The statistical method is the first approach in this study for constructing 3D microstructure. For this purpose, various appropriate filters should be used in the pipeline of the problem. These filters are shown in Figure 5.1. As it can be noticed, the most important filter which needs to be employed is “StatsGenerator” filter coming at the beginning of procedure.

Figure 5.1: Dream3D statistical approach filters and pipeline.

It should be noticed that, a successful pipeline requires appropriate filters in the right place. The utilized filters of the statistical pipeline used in this study are briefly introduced in the appendix. Only StatsGenerator filter as the most critical filter is discussed in this chapter, the rest filters can be found in the appendix.
This filter is the main filter in which the data of microstructural quantifications are inserted to define the material for the software. The first step in this filter is to define the existing phase within the material. By selecting adding a new phase icon, one can define crystal structure, fraction, type and name of each phase separately. Since in this study we deal with dual phase titanium alloy, $\alpha$ and $\beta$ phase with HCP and BCC crystal structures and fractions of 90% and 10% were implemented respectively. The window of StatsGenerator filter consisting of the defined phases are shown in Figure 5.2. Using the curve of feature ESD probability density function, one can define the grain size vs. its fraction in this filter. By using the grain size distribution obtained via EBSD method, the grain size of each phase was defined. For a better correlation, it seems better if the parameters of ESD probability density function is changed by the user in order to fit the real data of the grain size. “Mu” is the average value of the lognormal grain size distribution, “Sigma” is the standard deviation of the lognormal grain size distribution, “Sigma Cut Off Value” permits the user to tune the distribution to avoid very large or very small grains, “Bin Step Size” is defined as the size of bin to use for segregating the grain size distribution into classes for relatively connecting other statistics to predefined grain size. The locations and values of the mentioned parameters are shown in Figure 5.2. For the first 3D construction of the microstructure, phases can be assumed to be equiaxed or rolled type. In this study, both of the mentioned types are constructed which will be shown further.

Finally, to visualize the outputs of Dearm3D, Paraview software was used. “Xdmf” file of each volume microstructure, was imported and visualized. The results of statistical approach for equiaxed microstructure and rolled microstructure are illustrated in Figure 5.3. As it can be noticed, alpha and beta phases are distributed homogeneously throughout the 3D volume microstructure. In Figure 5.3(b), alpha and beta phases are demonstrated with blue and red colors respectively. The result of rolled microstructure is provided in Figure 5.4. Each phase exhibits specific orientation distribution based on the input data. On the other hand, one of the disadvantage of this approach could be the distribution of beta
Figure 5.2: StatsGenerator environment of Dream3D.

phase. As it was manifested in previous chapters, beta phase normally appears in the grain boundaries of alpha phase in equiaxed type. Whereas, there exists some beta phases within alpha grains in the resultant digital microstructure. To overcome this disadvantage and to be much more precise, the realistic approach of digital microstructure construction was utilized which will appear next. However, it is worth mentioning that, statistical approach is very convenient and useful by which various types of microstructure can be generated and further used in simulations without spending time and energy on experimental works to gain realistic data. Nevertheless, realistic data are essential for validation of the predictive modeling performed in any stage.
Figure 5.3: (a) Statistical 3D volume equiaxed microstructure, (b) Present phases.
Figure 5.4: Statistical 3D volume rolled microstructure.
5.2 Realistic approach

As it was mentioned, a more realistic digital microstructure construction was conducted by direct importing of EBSD results into Dream3D software. A new pipeline was generated by adding more essential filters from the toolbox of the software. Compared to statistical approach, some more filters were used in the pipeline illustrated in Figure 5.5(a). In order to extrude the surface microstructure to a 3D representation, the pipeline of Figure 5.5(b) was utilized.

![Realistic digital microstructure pipeline](image)

![Realistic 3D digital microstructure pipeline](image)

Figure 5.5: (a) Realistic digital microstructure pipeline, (b) Realistic 3D digital microstructure pipeline.

The main difference is to use “Copy Data Container” and “Append Z Slice” to copy the image and extrude it to obtain a thick 3D representation. The more accurate way would be cutting the workpiece in various sections, and combining the resultant EBSD data to construct the whole 3D microstructure of the sample.
The results of realistic microstructure construction are illustrated in Figure 5.6 and Figure 5.7 for S1 and S2 respectively. As it can be noticed, the generated volumes are very analogous to experimental data. Here, beta phases are also placed based on the EBSD data mainly at the boundaries of alpha phase. The final 3D digital microstructure of S1 is demonstrated in Figure 5.8.

Figure 5.6: (a) Realistic digital microstructure of S1, (b) Present phases.
Figure 5.7: (a) Realistic digital microstructure of S2, (b) Present phases.
Figure 5.8: Realistic 3D digital microstructure of S1.
Chapter 6

Conclusions

In this thesis, the process material interaction considering microstructural characteristics was studied in micro scale milling of Ti6Al4V titanium alloy. The machinability of various microstructures i.e. equiaxed plus elongated, enlarged equiaxed, lamellar, martensitic containing different grain sizes, grain morphologies, phase fractions/distributions was evaluated. Based on the obtained results, there exist both advantageous and disadvantageous associated with each microstructure in terms of machinability and process outputs. In case of cutting forces, lower forces were attained for harder microstructures with low ductility, specifically martensitic microstructure. As built-up edge (BUE) is concerned, the sample with large equiaxed grains exhibited lower amount of BUE which can be favorable. In term of surface quality, the as-received fine equiaxed plus elongated sample showed better surface in the same micro milling conditions applied on various samples. Regarding burr formation, martensitic microstructure resulted in lower amount of burrs after micro milling tests. Therefore, it is not possible to gain all the desired micro milling outputs in a specific microstructure. However, this study provided the detail analysis over effect of microstructure on process outputs by which one can decide on appropriate material condition for a desired purpose. Considering the findings of this study, by using a sample with enlarged grain size attained by appropriate heat treatment, it is anticipated that, less energy, tool damage cost, burrs and a good surface quality can be achieved.
However, if one considers the surface quality as the most critical desired output, lower grain size of the material will yield a better surface with low roughness. Moreover, the microstructural induced changes after micro milling were investigated. The as-received sample with fine equiaxed plus elongated grains endured dynamic recrystallization (DRX), while this phenomenon was not observed in within the microstructure of enlarged equiaxed specimen. The main conclusions of this thesis can be mentioned as follows:

- Differences in mechanical properties associated with different grain size, grain morphology and phase fraction within the microstructures affected the deformation behavior of the samples during micro milling.

- Smaller grain size (both $\alpha$ and $\beta$) and lower $\beta$ phase fraction led to higher cutting force in micro milling. Although martensitic microstructure revealed a high hardness value among the specimens, it generated lowest cutting force due to its low toughness and ductility.

- Microstructures of the samples significantly affected the built-up edge (BUE) formation in terms of shape and size. Lower grain size resulted in more BUE, which may have contributed to the increase in cutting forces.

- EBSD results revealed that micro milling experiments affected the microstructure of the machined samples. Dynamic recrystallization (DRX) occurred in the fine grain equiaxed sample at some specific feed rates.

- EBSD investigation indicated that up milling and down milling can result in different textures within the machined surface. In the micro milling experiment, a more compressive deformation occurred in the down milling stage compared to the up milling.

- Surface quality and amount of burrs of the micro milled workpieces were affected by various microstructures. Smaller grain size led to lower surface roughness and higher amount of burrs.
Recommendations for future works

- Microstructural induced changes by micro milling can also be extended to study on other types of Ti6Al4V microstructures including lamellar and duplex.

- A good idea would be investigating the influence of material microstructure on tool wear during micro scale machining processes.

- Transmission electron microscopy (TEM) can be utilized to gain further understanding toward the possible microstructural evolutions within the machined surfaces, BUEs, chips and burrs.

- A detailed study over the impact of microstructural anisotropy on process outputs of micro scale milling can also be carried out.

- Finally, the results of this thesis along with the generated digital microstructures can be implemented in computer simulations and microstructure based predictive modeling of micro scale milling process.
Bibliography


Appendix A

Dream3D information

The brief explanations of the utilized filters in Dream3D software in this study, are given in this part.

Initialize Synthetic Volume

This filter generates an empty volume made of an image geometry to function as a base for introducing features to produce a synthetic microstructure. In this filter, a data container was also generated to maintain the generated image geometry and a matching cell attribute matrix. The dimensions, resolutions and origins of the created 3D volume. This filter can estimate the number of grains may locate in the volume based on the statistical data implemented previously. Notice that, in “required objects” part of this filter, the data container generated in the previous filter was selected.

Establish Shape Types

Using this filter, the specific shape type to each phase was assigned. Ellipsoid, Super ellipsoid, cube octahedron and cylinder shape types can be selected for the grains. Here, ellipsoid shape was assigned for both alpha and beta grains. In the “required objects” part of this filter, phase type was selected to complete this section.
Pack Primary Phases

Basically, this filter is utilized in order to pack the existing phases in the synthetic microstructure volume. Also, the filter specifies the available volume for inserting the primary grains. Again, in the “required objects” part, the data needed for running this filter were selected from previous filters using statistics and phase data.

Find Number of Features

This filter was used in order to determine the number of features or grains. The generated phase were selected to complete this filter.

Find Boundary Cells (Image)

This filter was employed to define, the number of adjacent cells that are possessed by a different feature or grain for each cell. The feature number 0 was ignored in this section. Features Ids as the prerequisite of this filter were selected to complete this part.

Insert Precipitate Phases

If in the StatsGenerator section the second phase is manipulated, this filter inserts the second phase as precipitate in the volume by some predefined regulations. Feature Ids, phases, statistics, boundary cells, phase types, shape types and number of features were entered for this filter as inputs. If the “match radial distribution function” is filled in the filter, the software considers the data of radial distributions in StatsGenerator filter, which also takes some more time to perform.

Find Feature Neighbors

This filter specifies that which grains are in adjacent to a specific individual grain. Feature Ids and cell attribute matrix were imported to this filter by using the previous generated data.
Find Surface Features

Utilizing this filter, the grains showing at the surface of the microstructural volume were created based on the software directory.

Match Crystallography

Since alpha and beta phases exhibit different crystallographic lattice structure and orientation distribution functions (ODF), this filter was used to match these parameters. Again, note that all the required information need to be imported to complete this filter.

Write DREAM.3D Data File

In all types of Dream3D pipelines and in every problem, this filter is used to save the output of the all utilized filters. This filter stores the data with two main formats i.e. “dream3d” and “Xdmf”. The latter format is necessary to visualize the results of Dream3D by Paraview package.

Abaqus Hexahedron Exporter

In order to use the generated volume microstructure in ABAQUS software, this filter was used to export some critical files for further simulation goals. These outputs are as follows: example_nodes.inp, example_elems.inp, example.inp (the master file), example_elset.inp and example_sects.inp.