

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE
ENGINEERING AND TECHNOLOGY

**THE EFFECT OF SURFACE ROUGHNESS ON MECHANICAL BEHAVIOR
OF COMMERCIALLY PURE TITANIUM IMPLANTS PRODUCED BY
SELECTIVE LASER MELTING**



M.Sc. THESIS

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Department of Metallurgical and Materials Engineering

Materials Engineering Programme

JUNE 2018

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

**SEÇİCİ LAZER ERGİTME YÖNTEM İLE ÜRETİLMİŞ TİCARİ SAFLIKTAKİ
TİTANYUM İMPLANTLARIN YÜZEY PÜRÜZLÜLÜKLERİNİN MEKANİK
DAVRANIŞA ETKİSİ**

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HAZİRAN 2018

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To my family,



FOREWORD

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May 2018

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ABBREVIATIONS

2D	: Two-Dimensional
3D	: Three-Dimensional
AB	: As-built
AFM	: Atomic Force Microscopy
AM	: Additive Manufacturing
Ar	: Argon
ASTM	: American Society for Testing and Materials
AVG	: Average
B	: Back
CAD	: Computer Aided Design
CM	: Confocal Microscopy
CMF	: Cranio-Maxillofacial
cpTi	: Commercially Pure Titanium
et al.	: and others
F	: Front
GE	: General Electric
GPS	: Geometric Product Specification
ISO	: International Organization for Standardization
L	: Left
LD	: Left-Down
LU	: Left-Up
MPP	: Materialise Post-Processing
NV	: a Dutch term for a public limited-liability company
PP	: Post-processed
RD	: Right-Down
ROI	: Region of Interest
RU	: Right-Up
SB	: Sand-blasting
SEM	: Scanning Electron Microscopy
SLM	: Selective Laser Melting

SPM	: Scanning Probe Microscope
STD	: Standard Deviation
US	: United States
UTS	: Ultimate Tensile Strength
XCT	: X-ray Computed Tomography



SYMBOLS

μm	: Micrometer
Hz	: Hertz
kN	: Kilo Newton
log	: Logarithmic
min	: Minute
mm	: Milimeter
MPa	: Megapascal
°	: Degrees (deg)
P	: Primary profile
R	: Roughness profile
R-ratio	: Stress ratio for fatie (R)
Rt	: Total height
Rz	: Maximum height
S	: Areal parameters
T	: Thickness
Ti	: Titanium
V	: Volumetric parameters
W	: Waviness parameters
w	: width
Zp	: Maximum peak height
Zv	: Maximum valley depth
α	: Alpha
β	: Beta
Δ	: Delta



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THE EFFECT OF SURFACE ROUGHNESS ON MECHANICAL BEHAVIOR OF COMMERCIAL PURE TITANIUM IMPLANTS PRODUCED BY SELECTIVE LASER MELTING

SUMMARY

Implants produced by selective laser melting (SLM) have differentiating surface roughness caused by the process itself and applied surface treatments. Since surface quality and mechanical properties are critical parameters for implants and surface roughness is a known factor for stress concentration, it is aimed to investigate the effect of surface roughness on commercially pure titanium implants produced by SLM.

Surface roughness is affected by several process parameters but this study focuses on the effect of position and orientation on surface roughness.

First step is specified as determining the roughness measurement method ideal for this work. Therefore, 3 different roughness measurement methods as in confocal microscopy (CM), tactile profilometer and scanning electron microscopy (SEM) are compared by using the most common surface roughness parameter, Ra.

Secondly, in order to determine the effect of position and orientation on surface roughness and determine roughness ranges across the build chamber, a build is designed with parts at 15 different positions with 6 different orientations that are specified considering the location of laser.

Finally, for mechanical characterization, tensile bars are designed considering both the standards stated for tensile and fatigue test. Some parts are also post processed to see the post processing effect. For every position, 5 samples are subjected to tensile testing with 5 MPa pre-load until the part failure so that the average yield stress and UTS for every position are determined.

Calculated average yield stresses are used to calculate fatigue test input. 5 stress levels are determined and 2 samples for low cycle, 3 samples for high cycle fatigue data are tested with load controlled, tension-tension fatigue test set-up with 60Hz frequency and R of 0.1.

Surface finishing is differentiated by MPP and SB in addition to as-built form. Some samples are post processed with the standard post process of Materialise. Sand blasting and anodization are applied while for SB samples only sandblasting is applied. Then samples are tested to see pp effect on surface roughness and mechanical behavior.

When the results are analyzed considering the roughness range and sample amount, the ideal roughness measurement method is determined as tactile profilometer because of its capability, repeatability, practical application and effectiveness if time and cost.

The roughness range across the build plate is quantified for different positions and measured Ra values are in the range of 7 to 24 μm across the build plate. It is also concluded that at the right bottom side of the build chamber, and for the orientations perpendicular to the laser beam, surface roughness increases.

Relation between Rz and thickness is specified and an equation is suggested to eliminate the effect of roughness on thickness. Since the thickness is effective on cross section calculations used for mechanical characterization, suggested equation is used to recalculate stress values measured with tensile and fatigue tests.

Even though measured tensile and fatigue results indicate that increased surface roughness has a negative effect on tensile strength and fatigue life, recalculated tensile and fatigue results display no difference occurs with varying surface roughness. Therefore, it is shown that surface roughness has an effect on thickness hence has an effect on cross section that is affecting fatigue and tensile test results but it does not have a real significant effect on mechanical behavior of SLM printed cpTi parts.



SEÇİCİ LAZER ERGİTME YÖNTEMİ İLE ÜRETİLMİŞ TİCARİ SAFLIKTAKİ TİTANYUM İMPLANTLARIN YÜZEY PÜRÜZLÜLÜKLERİNİN MEKANİK DAVRANIŞA ETKİSİ

ÖZET

Bu proje, seçici lazer ergitme yöntemiyle üretilmiş ticari saflıktaki titanyum implantların yüzey pürüzlülüklerinin mekanik davranışa etkisinin incelenmesini kapsamaktadır.

Seçici lazer ergitme (SLM) son yıllarda kullanımı yaygınlaşan bir eklemeli üretim yöntemidir. Çağın getirdiği insan hayatını etkileyen yeni ihtiyaçlar ve gelişen teknoloji eklemeli üretimin keşfine ve yaygınlaşmasına sebep olmuştur. Eklemeli üretim yöntemi metaller, seramikler, polimerler gibi birçok malzeme için uygun olmakla birlikte, otomotiv, uçak-uzay, enerji ve sağlık sektörlerinde kullanılabilmektedir. Seçici lazer ergitme, bilgisayar destekli tasarımla (3 boyutlu modelleme) hazırlanmış karmaşık şekilli parçaların, toz hammaddenin lazerle katman katman ergitilip birleştirilmesi ile üretilmesi olarak tanımlanır.

Geleneksel üretim yöntemlerine göre birçok avantajı vardır. Öncelikle kompleks şekilli tasarımlar başta olmak üzere, parçaların üretim sonrası işlenme ihtiyacını minimuma indirdiği için zaman ve maliyet anlamında tasarruf sağlar. Ayrıca üretilen parçalar yüksek yoğunluklu dolayısıyla yüksek mekanik özelliklidir. Son olarak, özellikle sağlık sektörüne hizmet açısından en önemli avantajı, hastaya özgü detaylı implant tasarımlarının üretimini mümkün kılmasıdır. Özellikle medikal implant üretiminde titanyum ön plana çıkmaktadır. Ticari saflıktaki titanyum (cpTi) ve titanyum alüminyum alaşımları (Ti6Al4V) kemikle uyumlu mukavemetleri, şekillendirilebilirlikleri ve korozyon direnciyle en çok tercih edilen malzemelerdir.

Seçici lazer ergitme yönteminin önemli dezavantajlarından biri ise yüzey kalitesindeki değişkenliktir. Eklemeli üretim yöntemiyle üretilen parçalar, bu üretim şeklinin içsel özelliklerinin (katman katman üretim sonucu merdiven adımı etkisi ve lokal ergitme sonucu oluşan yüzey gerilmeleri sebebiyle topaklanma) etkisiyle düşük yüzey kalitesine sahip olabilir. Üretim parametreleri, kullanılan tozun özellikleri ve son ürün için planlanmış üretim sonrası işlemler (kumlama ve anodizasyon), seçici lazer ergitme ile üretilen ürünlerin yüzey kalitesini etkilemektedir.

Belli orandaki yüzey pürüzlülüğünün implantlarda osseointegrasyonu kolaylaştırdığı bilinse de, ön gözlemler sonucu özellikle aynı üretim alanından çıkan bazı parçaların sahip olduğu yüzey pürüzlülüğünün istenenden fazla olduğu farkedilmiştir. Ön testler sonucunda edinilen izlenim ise artan yüzey pürüzlülüğüyle gerilme direncinde düşüş olarak tanımlanmıştır.

Bu nedenle, bu tez kapsamında toz özellikleri ve lazer parametreleri sabit tutulup, yalnızca parçaların üretim alanındaki değişen pozisyon ve oryantasyonun yüzey pürüzlülüğüne etkisi ve belirlenen yüzey pürüzlülüğünün parçanın mekanik davranışı üzerindeki etkisi incelenmiştir.

Bu doğrultuda ilk adım yüzey pürüzlülüğünün tanımlanması olarak belirlenmiştir. Yüzey pürüzlülüğü yüzey dokusunun karmaşık elementlerini ayırt edebilmek için çeşitli parametrelerle tanımlanmıştır (Bu çalışmada Ra, Rz ve Rt kullanılmıştır). Yüzey pürüzlülük parametrelerini ölçmek için kullanılacak en uygun yöntemi seçmek üzere 3 farklı yüzey pürüzlülüğü ölçüm yöntemi karşılaştırılmıştır. 3 yöntem; temassız, temaslı ve nitel metod olarak sırasıyla Konfokal mikroskobu (CM), dokunsal profilometre ve taramalı elektron mikroskobu (SEM) olarak belirlenmiş, karşılaştırma yapılırken, en yaygın yüzey pürüzlülük parametresi Ra kullanılmıştır.

Daha sonra, parça yüzey pürüzlülüğünün üretim alanındaki pozisyon ve oryantasyona bağlı olarak gösterdiği değişim incelenmiştir. Bu amaçla lazerin konumu göz önünde bulundurularak, 15 farklı pozisyon ve 6 farklı oryantasyonla numuneler üretilmiştir. 15 pozisyon şu şekilde belirlenmiştir; bir tanesi lazerle aynı lokasyonda olacak şekilde tanımlanırken, diğer 14 pozisyon üretim alanının sağ ve soluna eşit olarak dağıtılmış ve pozisyonlar 1 den 6 ya kadar numaralandırılmış, numaradaki artış lazere olan uzaklığın artışı ile eşleştirilmiştir. 6 oryantasyon ise numunelerin z ekseninde rotasyonu ile ve açılı üretim yapılarak sağlanmıştır.

Son olarak mekanik karakterizasyon için, hem çekme hem yorulma deney standartlarına uygun çekme testi numunesi tasarlanmıştır. Üretim sonrası işlemin etkisini görmek için ayrılmış parçaların dışında her parça işlenmemiş haliyle kullanılmıştır. Çekme testi için her gruptan 5 numune 5 MPa ön yükleme ile parça kopana kadar test edilmiş, akma dayanımı ve nihai çekme mukavemeti değerlerinin ortalaması alınarak, o gruba ait ortalama değerler belirlenmiştir.

Hesaplanan ortalama akma dayanımı değerleri yorulma testi girdilerinin hesaplanması için kullanılmıştır. Yük kontrollü, gerilmeli yorulma testi 60 Hz frekansla uygulanmış, R değeri 0.1 ve sonsuz ömür 1 milyon çevrim olarak belirlenmiştir (implantlar için standart çevrim sayısı). 5 stres düzeyi belirlenmiş ve düşük çevrim değerleri için 2 şer, uzun çevrim değerleri için 3 er numune test edilmiştir.

Projenin ortaklarından Materialise adlı implant şirketinin implantlara standart olarak uyguladığı son işlemlerin yüzey pürüzlülüğü üzerindeki etkisini görmek için bir grup numune bu işleme tabi tutulmuş, bir grup numuneye ise yalnızca kumlama uygulanmış ve mekanik testler yapılmıştır.

Sonuçlara bakıldığında, yüzey pürüzlülüğü ölçme yönteminin belirlemesi kısmında, farklı yöntemlerle ölçülen değerler analiz edildiğinde, test edilen numunelere uygun kesme uzunluğu (yüzey pürüzlülüğü olarak sayılacak düzensizliğin frekansını belirleyen uzunluk) 2.5 μm ve 8 μm olarak belirlenmiştir. Temassız, temaslı ve nitel metodlar erişilebilirlik, ölçüm kapasitesi, tekrarlanabilirlik, doğruluk, maliyet ve zaman verimliliği bakımından değerlendirilmiştir. SEM ile ölçülen değerler küçük ilgi bölgesi (small region of interest) sebebiyle aralık dışı kalırken, CM ve dokunsal profilometre sonuçları birbiriyle uyumlu çıkmıştır. Bu proje kapsamında incelenen yüzeylerin pürüzlülük menzili ve numune sayısı da göz önünde bulundurularak, en uygun yöntem dokunsal profilometre olarak belirlenmiştir.

İkinci adımda lazer lokasyonu göz önünde bulundurularak, farklı pozisyon ve oryantasyonlarla üretilen numunelerin dokunsal profilometre ile pürüzlülük değerleri ölçülerek üretim alanındaki yüzey pürüzlülüğü menzili minimum 7 μm and maksimum 24 μm olarak ölçülmüştür. Özellikle numunelerin lazere dönük olmayan yüzeylerinde, üretim alanının sağına doğru gidildikçe yüzey pürüzlülüğünde artış

gözlemlenmiş, maksimum yüzey pürüzlülüğü üretim alanının sağ alt kısmında 24 µm olarak ölçülmüştür.

Farklı oryantasyonların etkisi incelendiğinde ise, lazer ışınına dik alan numunelerin lazere dönük yüzeylerinde, yüzey pürüzlülüğünün daha düşük olduğu gözlemlenirken, lazer ışınına paralel olan oryantasyonlarda, ön ve arka yüzeylerin pürüzlülük değerlerindeki farklılığın minimum olduğu gözlemlendi.

Bu veriler göz önünde bulundurularak, tezin üçüncü kısmında mekanik davranışın incelenmesi için üretilecek numuneler için 5 lokasyon (minimum pürüzlülük değerine sahip olan lokasyondan, maksimum değere doğru) ve tek oryantasyon (lazere dönük olan ve olmayan yüzey arasındaki farkın en büyük olduğu oryantasyon) belirlendi. Mekanik testler öncesi numunelerin yüzey pürüzlülüğü, kalınlık ve genişlik ölçümleri yapıldı. Maksimum yüzey pürüzlülüğünün (R_a) 20 µm, minimum yüzey pürüzlülüğünün ise 10 µm olduğu görüldü. Ayrıca, pürüzlülük artışıyla yüzey pürüzlülüğünün (R_z) doğru orantılı olduğu gösterildi. Bu bağlantı doğrultusunda, yüzey pürüzlülüğünün kalınlık üzerindeki etkisini kaldırmaya yönelik bir formül tanımlandı. Numunelerin ön ve arka yüzeylerine ait R_z değerleri toplanıp, kalınlık değerinden çıkarıldığında, yüzey pürüzlülüğünün kalınlık ölçümü ve dolayısıyla kesit alanı hesaplamalarındaki etkisinin kaldırılabilceği önerildi.

5 pozisyon için ölçülen ortalama akma dayanımı değerlerine bakıldığında, minimum pürüzlülük pozisyonunda 493 MPa, maksimum pürüzlülük pozisyonunda ise 448 MPa olduğu görüldü. Fakat önerilen formül ile kesit alanı değeri değiştirilip, stres değerleri tekrar hesaplandığında, hem işlenmemiş hem de üretim sonrası işlem görmüş numunelerde, farklı pozisyonların ortalama akma değerleri arasındaki farkın göz ardı edilebilecek kadar küçük olduğu gösterildi.

Çekme testinden elde edilen akma mukavemetinin %80'i kullanılarak maksimum pürüzlülüğe sahip grupla yorulma deneylerine başlandı. Farklı yüzdeler kullanılarak 5 stres düzeyi belirlendi. Numunelerin pozisyon farkına rağmen yakın akma dayanımına sahip olması göz önünde bulundurularak, ve karşılaştırma yapabilmek için, her gruba aynı stres düzeyleri uygulandı. Önerilen formül kullanılarak hesaplanan "pürüzlülükten arınmış kalınlık" değerleri ve yorulma testi sırasında uygulanan yük kullanılarak stres yeniden hesaplandı. Sonuçlar karşılaştırıldığında, yorulma eğrilerinin çakıştığı görüldü. Maksimum ve minimum yüzey pürüzlülüğüne sahip gruplar arasında kayda değer bir farklılık görülemeyince, ara değer gruplarına yorulma testi uygulanmadı. Yalnızca, üretim sonrası işlem görmüş numunelerde, önerilen formül kullanılıp, stres tekrar hesaplanırsa da yorulma eğrileri işlenmemiş numunelere göre farklılık göstermeye devam etti. Bunun sebebinin ise üretim sonrası işlemler dolayısıyla yaratılan kalıntı gerilmeler (kumlama işlemiyle) ya da sert oksit tabakası (anodizasyon işlemiyle) olabileceği düşünüldü.

Ayrıca maksimum ve minimum pürüzlülük pozisyonlarından alınan numunelerin mikroyapıları incelendi. Her iki grup için de $\alpha+\beta$ yapısı gözlenmiş, hızlı soğuma sonucu oluşan iğnemsî yapının hakim olduğu görülmüştür.

Sonuç olarak, yüzey pürüzlülüğünün SLM yöntemiyle üretilen cpTi numunelerin mekanik davranışları üzerinde bir etkisi olmadığı gösterilmiştir.



1. INTRODUCTION

Over the past few decades, the increasing demand in amount and qualification of human needs and altering technology bring on a new production technique; additive manufacturing (AM). Among many different application areas like aerospace, automotive, consumer goods, medical industry has also embraced additive manufacturing techniques.

Selective laser melting (SLM) is one of the mostly used AM methods for satisfying the property requirements of advanced medical applications. SLM enables the production of complex shapes and internal features which are limited in the case of conventional manufacturing methods. Also it is applicable to several kinds of materials. Another advantage of SLM is its ability to reduce the lead time of products. Finally, customization is an important advantage of SLM especially for the manufacture of patient specific implants [1].

SLM is defined by Kruth et al. as “a powder based AM method in which a complex three-dimensional part is built by melting and solidifying the required geometry, layer by layer, on top of each other” [2]. After one layer is completed, the platform moves down in the z-axis and another layer of powder is scanned and melted onto the previous layer [3]. Intrinsic properties of this layer-by-layer production make the process limited in terms of surface quality [4].

Surface quality of parts produced via SLM are affected by several variables such as the process parameters, post-processing or the properties of the starting powder.

There are many studies examining the effect of process parameters on parts properties like porosity, roughness and strength. Kleszczynski et al. stated that even when all process parameters like scan parameters or powder size and distribution are fixed, the position and orientation of the product inside the build chamber still have an effect on surface roughness [5].

Controlled surface roughness is critical for implants in terms of quality. Apart from its positive biological effect on osseointegration, surface roughness has also an effect

on implant failure [6]. Several studies show that roughness affects mechanical properties [4, 6, 7, 8]. Specifically, because of possible notching effect on the surface, fatigue life shows variety [6, 7].

1.1 Problem Statement

One of the ways of enhancing the mechanical performance is to alter surface properties. It is known that surface properties are related to the production process and they are also key parameters for high performance [9]. Therefore, getting the information regarding surface properties by characterization can lead for not only prospering production but also satisfying performance for medical implants.

Implants produced by SLM have differentiating surface roughnesses caused by the process itself and applied surface treatments. Moreover, it is reviewed that surface roughness is affecting mechanical properties.

However, to the best of knowledge of the author, the effect of roughness on mechanical behavior of commercially pure titanium (cpTi) implants produced by SLM is an underexplored topic.

Since surface quality and mechanical properties are critical parameters in medical implant applications, this study aims to further investigate the correlation between roughness and mechanical behavior of cpTi cranio-maxillofacial (CMF) implants produced by SLM for assessing the ultimate aim of improved implant quality and manufacturing process. In this study, after the ideal method for determining the surface roughness of the products will be selected, the effect of orientation and position in the process chamber will be determined and the effect of post processing will be examined.

1.2 Aim and Objectives

1.2.1 Aim

The aim of this master thesis is split into three parts which are defined as; determining the ideal roughness measurement method for roughness characterization of cpTi parts for CMF applications produced via SLM, quantifying the variation in roughness across the build chamber and applying mechanical tests to correlate these results with the roughness values measured with the determined technique.

Firstly, for the purpose of roughness characterization, the first step is roughness measurement [9]. Bhushan stated that roughness can be defined as unevenness or variations in the height of the surface. The roughness measurement techniques can be grouped into two as contact or non-contact types [10]. Tactile profilometer as a contact type roughness meter is one of the oldest, cheapest and most accurate contact techniques available [11]. In literature, for measuring roughness of SLM processed Ti6Al4V, Focus variation microscopy, Atomic force microscopy (AFM), Confocal microscopy (CM) and X-ray computed tomography (XCT) techniques have been used as non-contact types [10, 12, 13]. In addition to these quantitative techniques, optical microscopy and scanning electron microscopy (SEM) can be used for getting qualitative information.

For the first part of this thesis, amongst several methods, a quantitative roughness measurement method will be selected based on a test method capability v. cost assessment.

As it is mentioned before, since both the orientation and position in chamber are known to have an effect on roughness, the second part is to determine the roughness variation across the chamber. When the range of roughness of the parts according to regions, and the effect of post-processing on these ranges are determined, post processing of parts can be specified according to their initial roughness. However, optimization of roughness or post process is out of the scope of this thesis.

The final part of the thesis aims to determine the effect of surface roughness on mechanical behavior. After printing and post-processing where applicable, the mechanical properties of these different types of samples will be assessed.

1.2.2 Objectives

The theoretical background and literature overview about SLM, post processing, roughness and its measurement will be completed.

Experiments will be performed with available surface roughness measurement methods; tactile profilometer, SEM, Confocal microscopy results will be compared and the most suitable method for this study in terms of engineering, time and cost will be selected.

For the second part, the design of the build with the consideration of creating samples that have the same orientation but different positions within the build chamber will be completed. Fully dense parts will be built within the same batch with the same scanning parameters. Roughness range will be quantified by the chosen technique. The effect of position in the chamber will be determined by comparing samples that have the same orientation and samples with different orientations at the same positions will be examined to determine the effect of orientation.

Afterwards, the effect of roughness on mechanical properties will be assessed. Surface roughness will be varied by post processing. In order to see the difference between different surface roughness values, tensile and fatigue tests will be applied to these as built and post processed samples.

1.3 Hypothesis

If the effect of processing conditions during SLM, on the final parts surface roughness is fully understood, these roughness values will then be correlated to the mechanical behavior of components and post processing steps can then be optimized to try to counteract the effect of processing conditions inherent to the SLM process.

1.4 Outline of Thesis

The following chapters discuss about the effect of surface roughness on mechanical behavior. Chapter 2 will consist of an in depth literature review covering the main principles of SLM and roughness measurement techniques and those employed in SLM. There will be a brief discussion for additive manufacturing in terms of Selective Laser Melting, its advantages and challenges will be mentioned. Afterwards, the properties of Titanium will be discussed shortly since the focus of this thesis is cpTi. Finally, a roughness definition and detailed information of roughness measurement principles and methods will be given. At the end of this chapter, the methods to be applied are defined. Chapter 3 presents the material and methods, the equipment and method details will be given. After material information and SLMpart production is described, experimental outline will be stated and methods of roughness measurement, post processing and characterization are

determined. In Chapter 4, results will be shown and discussed in three parts. Firstly results for comparing different roughness measurement methods will be given and an optimal method will be selected. Secondly, roughness measurement results across the build plate will be determined and in the last subsection, microstructural and mechanical characterization results will be given. Finally Chapter 5 will conclude this thesis work and future work will be defined.





2. LITERATURE REVIEW

2.1 Introduction

In this section, there will be an explanation of the SLM process and roughness formation during SLM. Terms of orientation and position in the chamber will be discussed in detail and post processing steps will be introduced. Afterwards, general information about Titanium for implant applications and properties of cpTi will be stated. Finally, a definition of roughness and roughness measurement principles will be given.

2.2 Selective Laser Melting

SLM is a powder based additive manufacturing method. Kruth et al. have defined SLM as; “a powder based AM method in which a complex three-dimensional part is built by melting and solidifying the required geometry, layer by layer, on top of each other” [2].

Since it has been discovered in 1995, it is widely used in different industries like aerospace, energy, automotive and medical. These are industries where typically either mass customization is a requirement or hard to process alloys by conventional techniques are used.

The most common applications of SLM for the medical industry are surgical implants and dental applications [15]. For these applications, the most commonly used titanium powders are Ti6Al4V and cpTi (will be discussed in section 2.3 in detail) [16].

When it is compared to the conventional methods such as investment casting for aerospace applications or directly machining a final component from a bulk material, SLM has several advantages. Firstly, SLM enables reduction of production steps which also reduces time-to-market and cost [1]. Having a wide spectrum of materials that can be used is another advantage of the process. Maraging steels, aluminum-, titanium-, cobalt-chromium and nickel- based alloys can currently be produced by

SLM. Furthermore, SLM enables high density, close to 100%, which means high mechanical properties that is equivalent or even better than the conventional methods [14]. Finally, another significant advantage of SLM is the elimination of shape restrictions. It enables production of complex shapes and “patient specific customization” for medical applications [17, 18].

The working principle of SLM is illustrated in Figure 2.1. After the computer aided design (CAD) model is completed, it is sliced into layers and transferred to the SLM machine. Typically with an SLM machine you will find a build chamber with a build plate, pistons, powder supply and a laser as you can see in Figure 2.2. In order to build the model, firstly, a powder layer is spread onto the build platform. Then, geometrical information of the first layer is transmitted from CAD to the laser beam. Since titanium is highly reactive to the interstitial elements like oxygen, nitrogen, carbon and hydrogen, powder is scanned under an inert argon atmosphere. Though, it is stated that because of solution strengthening, some amounts of oxygen can be used to enhance mechanical properties [19]. Selectively melted powder creates a melt pool to be consolidated in order to produce the first layer. After the first layer is scanned, the working platform is lowered by one layer thickness in order to spread a new powder layer for scanning the next slice. Spreading, fusing and lowering steps are repeated until the part is completed [14, 17, 20].

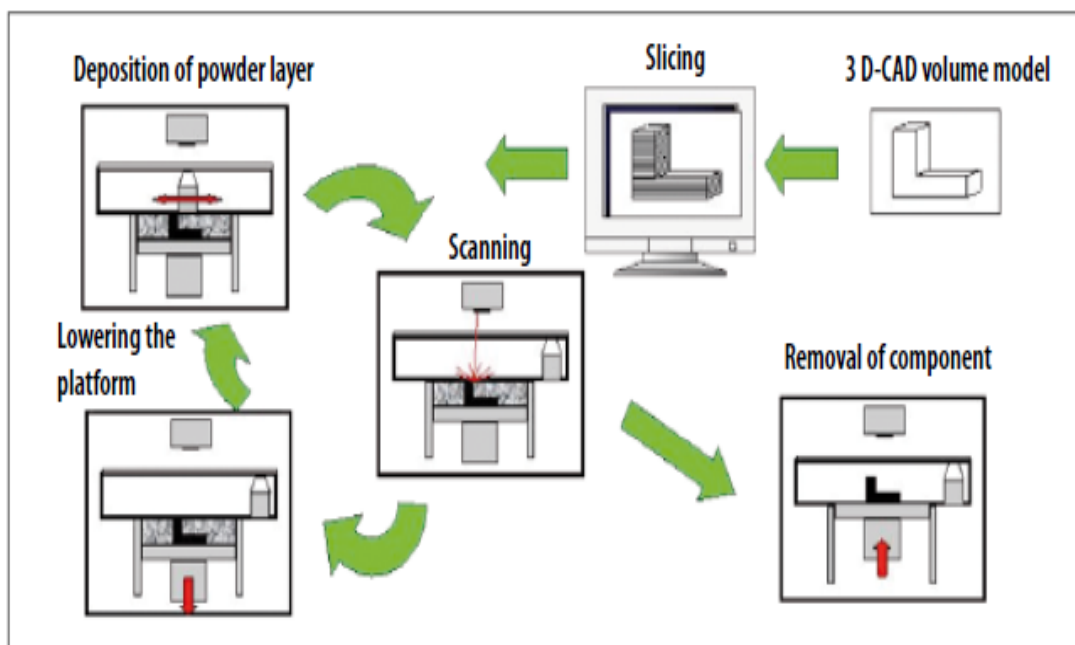


Figure 2.1 : Working principle of SLM [14].

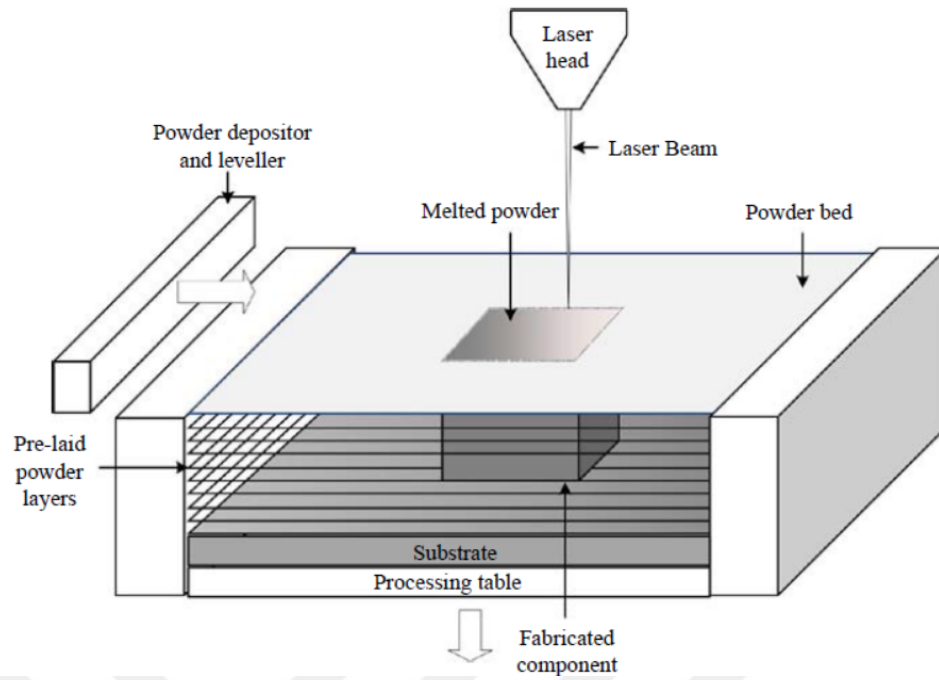


Figure 2.2 : Presentation of SLM build chamber and printing mechanics [3].

While designing the SLM process, in addition to the basic design which includes design of minimum wall thickness, maximum length-to-height ratio, holes and internal channels etc., part orientation should also be designed carefully for quality and cost.

Part orientation affects build time, quantity of supports (removal of which should not be causing difficulty), residual stresses and surface roughness.

Part orientation is defined by overhang and support terms. Overhangs are natural outcomes of layer-by-layer production. And the angle between build platform and part is defined as overhang angle as shown in Figure 2.3. If the overhang angle is not too low, the previously melted layer will support the next one and extra support requirement will decrease. It is stated that overhang angle should be a minimum of 45° to avoid using support structures. In Figure 2.4, it can be seen that overhang area is smaller at 45° than 90° .

Further, since SLM includes local melting and rapid cooling, stress is generated on SLM processed parts. Along with the loose powder base of the process, parts are easily affected by this stress and without the absence of supports, they curl up or agglomerate loose powder particles around the focal point. A curled layer would restrain the next layer, therefore a mistake in part orientation design can cause build failure [15].

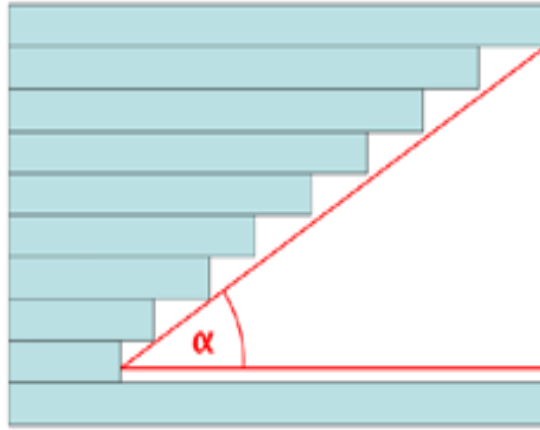


Figure 2.3 : A visual representation of an overhang angle α [15].

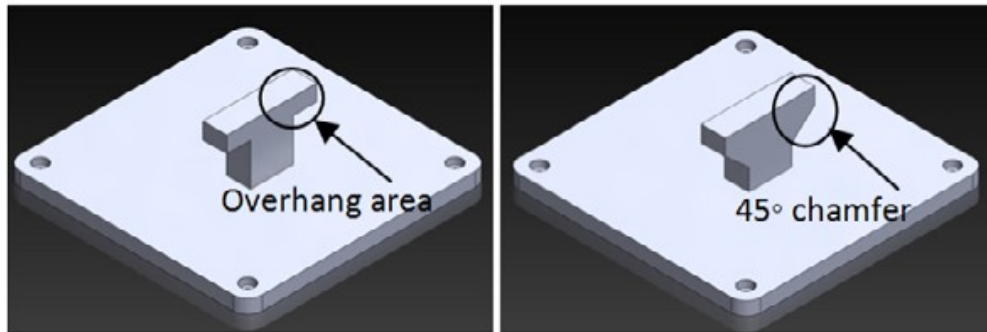


Figure 2.4 : a) Angles of 90° having a larger overhang area, b) 45° chamfer that requires less supports [15].

2.2.1 Process parameters

Determining process parameters is another important step for process design. Laser-material interaction during the process affects the properties of the printed parts. Since there is a short interaction between the energy of laser beam and powder bed, issues caused by temperature gradient can be seen in SLM. In other words, cooling rates are affected by parameters and they formalize the microstructure of the parts. Also with the additional effect of gravity and capillary forces, directional solidification creates a texture on the parts. Consequently, mechanical properties differ according to the interaction. Therefore, in order to have a qualified part, process should be optimized. Otherwise, potential defects like porosity, balling, residual stress or cracks can occur [21].

There are many different process parameters for the control and optimization of the SLM process. For example, Srivatsan et al. declares that the powder size, shape and size distribution has an effect on density, it is stated that finer particles in spherical shape indicate higher density of the final part [21].

Also, scan parameters as can be seen in Figure 2.6 that are defined as laser power, scanning speed, layer thickness and scan line spacing should be optimized to produce high quality parts.

Kruth et al. (2010) presented in the literature that if scan speeds are sufficiently low and satisfactory interaction is ensured, almost independently of the layer thickness, high density parts can be achieved. However, at higher scan speed values, it is observed that in order to get higher density, layer thickness should be decreased [22].

Yadroitsev et al. (2011) analyzed the influence of the hatch distance (defined as shift between tracks in the plane of scanning (See also Figure 2.6) on morphology of the first layer and surface structure. They processed stainless steel powders with SLM with varying hatch distance from 60 to 280 μm and observed that decreasing hatch distance, increase the time for heating for side regions and creates lower tracks. In order to be able to produce a smooth surface the maximum hatch distance should be calculated considering the average width of elongated lines of molten powder scanned by laser beam [18]. Also, they emphasize the effect of laser power and scan speed on irregularity or balling.

Another aspect affecting parts' properties like density, residual stress and microstructure is scan pattern. For example, Thijs et al. state that rather than unidirectional, using alternating hatch directions as scanning strategy enables not only a more isotropic structure but also a higher density [23].

Yadroitsev et al. (2009) declared that regarding the scanning directions as in horizontal or vertical, anisotropy of mechanical properties is observed. When average values of Young's modulus for the horizontal and vertical samples are compared, horizontal samples happened to have much higher values than the vertical ones. This is explained by the relatively higher amount of thermal stress accumulated corresponding to a greater number of layers of vertical samples [24]

Finally, orientation and position in the chamber must be considered while designing the build. Kleszczynski et al. (2015) proved that there is difference in surface roughness values for 9 different positions and two surfaces of the samples as in inner and outer surface (as shown in Figure 2.5).

It is stated that outer surface have higher surface roughness than the inner surface. Also, it is observed that surfaces that face the borders of the build chamber compared

to other orientations have higher outer roughness, whereas the sample in the middle show quite uniform outer roughness. Also, the roughness values for the right side are slightly higher than the left side of the chamber. It is also emphasized that, more decisive information about the resulting mechanical properties is required for the practical usage of SLM in everyday production [5].

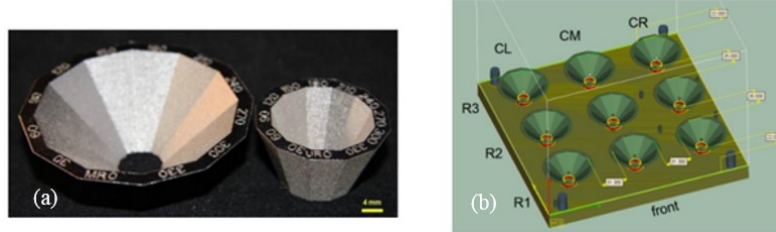


Figure 2.5 : a) Sample on the left with the overhanging angle of 45 degrees and sample on the right with the overhanging angle of 70 deg; both split into 30 deg slices to examine different z-rotations. b) 9 different locations defined across the chamber [5].

Another study about position and surface roughness in SLM is done by Hitzler et al., it is stated that surface roughness of the samples closer to the door side of the chamber is higher than the ones closer to the back side of the chamber. Also they stated that sides facing the gas flow and sides averting the stream does not show a difference worthy of note [25].

All above parameters (see also Figure 2.6) are affecting microstructure, texture and mechanical properties of the parts [21]. Yet, there is no definite way defined for complete process control of SLM [26]. In this study, to eliminate the effect of other parameters and to observe the effect of position and orientation in the chamber, everything but position and orientation will be stabilized for the build.

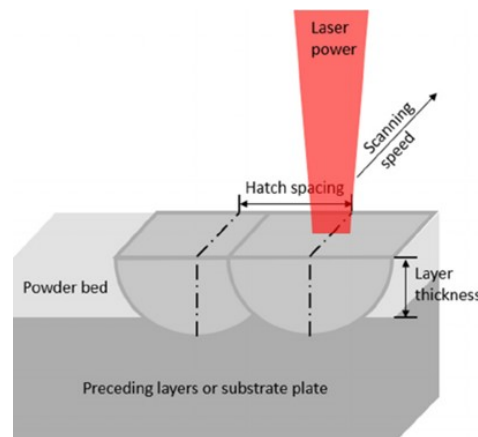


Figure 2.6 : SLM input parameters: laser power, scanning speed, hatch spacing, and layer thickness [27].

2.2.2 Post processing

Post processing is applied after removing the parts out of the build chamber to enhance their mechanical properties and give the final shape.

Firstly, removal of powder and support structures is completed. Then, heat treatment is applied for several purposes. These reasons can be listed as; improving general mechanical properties such as hardness or ductility, stabilizing the microstructure in the form that enhances fatigue performance or creep resistance and finally, for relieving residual stress that has occurred because of temperature gradients. However, it should be noted that the heat treatment step is not suggested for every material used in SLM. For example, even heat treatment enhances mechanical properties of Ti6Al4V, it is stated that it causes a decrease in hardness of SLM produced AlSi10Mg [21, 28].

After heat treatment, surface finishing and cleaning are important for SLM produced parts, especially for medical applications. Surface cleaning can be done by chemical or electrochemical treatments. Since there is a possibility of particle migration into the body from the implant, it must be ensured that there are no trapped particles that can migrate in cavities or pores [21, 28].

Also, surface roughness should be appropriate for implants (will be discussed in detail in Section 2.3.1). Surface treatments that are being applied on Ti implants to create controlled surface roughness can be grouped as mechanical (machining, polishing, blasting), chemical (solvent cleaning, wet chemical etching), electrochemical (electropolishing, anodizing) or thermal and laser methods (selective laser erosion) [29, 30].

Sand blasting is a mechanical surface treatment mainly used to remove particles from the surface due to being a line of sight technique and makes the surface smoother [27].

According to the study of Mertens et al., the main advantage of sandblasting after SLM, as can be seen in Figure 2.7, is increased density of the structure because it removes the weak upper layers. However, it is difficult to apply sandblasting perfectly on the corners of overhang structure and it causes a curved surface [31].

Anodization on the other hand is applied to increase corrosion resistance of implants by creating a homogenous TiO₂ layer on the surface [32]. It also increases bioactivity

of the implant. Sul et al. stated that there is a significant effect of anodization on roughness of pure titanium implants [33].

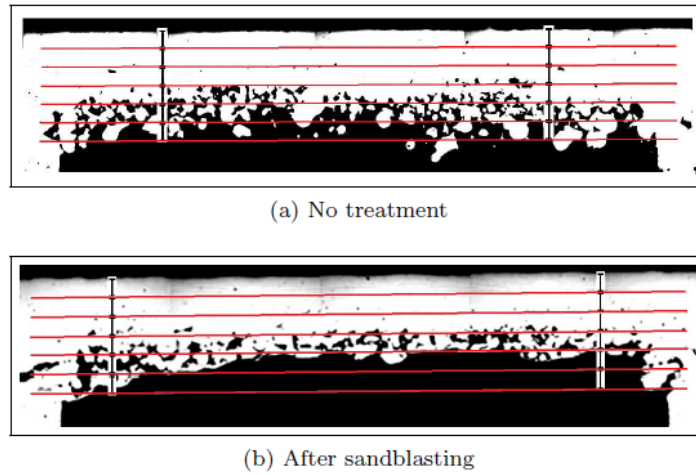


Figure 2.7 : Microscope image of overhang structure showing the effect of sandblasting on density.

2.2.3 Roughness and SLM

The causes of surface roughness occurrence in SLM can be grouped into two.

Firstly, as a consequence of poorly controlled layer-by-layer production and intrinsic properties of SLM, a problem arises: Poor surface quality of the parts that are not comparable to the parts' produced with conventional methods [34, 35]. The intrinsic properties of SLM as in; “stair step effect”, “balling effect”, unequal heat dissipations can be considered as natural resources of surface roughness [34]. Firstly, because of layer by layer production, the stair step effect occurs as can be seen in Figure 2.8. In Figure 2.9, surface roughness occurrence by stair step effect is illustrated. Another effect that is causing roughness is balling of molten metal which is defined as the formation of spherical structures due to the surface tension between molten powder and substrate (increased by low scan speeds and high repetition rates) [3,34,36]. Also, gravity is effective on the difference in upward and downward facing surface roughnesses. Co-occurrence of unstable melt pool and molten powder makes unsupported melt pool dips into the unmelted powder below. Finally, because of the difference in heat transmission capabilities of powder and molten metal, thermal gradients occur and cause partially melted and bonded particles at the edges [34, 37].

Apart from natural roughness, there is also roughness created by surface treatments that are affecting surface morphology like sandblasting and anodizing [31, 38].

As also discussed in section 2.2.1, there are a lot of studies examining the effect of parameters on roughness like powder particle size, layer thickness, laser and scan parameters, scan strategy, the orientation of the component in the building chamber and surface treatments. Further, those parameters affect mechanical properties of the parts built by SLM.

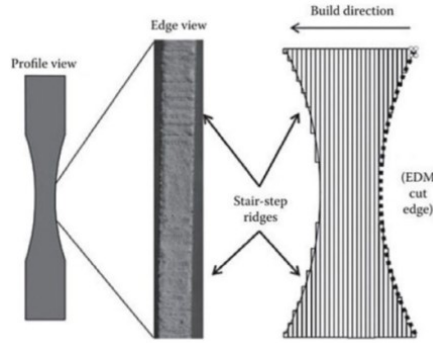


Figure 2.8 : Stair step effect in inclined surfaces of selective laser melted Ti6Al4V samples [36].

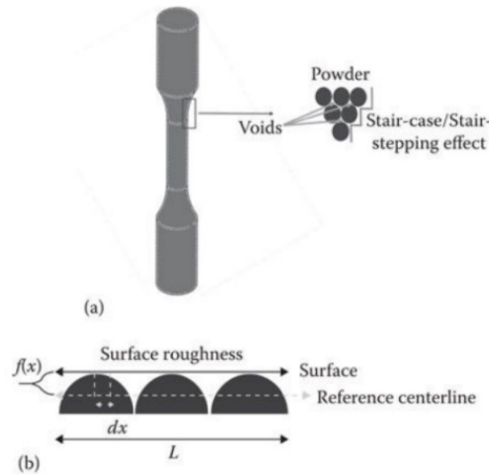


Figure 2.9 : a) Schematic of stair step effect b) schematic to determine surface roughness [36].

2.3 Titanium for Medical Implants

Titanium is used in many medical applications like, artificial joint replacement, maxillofacial reconstruction or audiological applications. Adequate mechanical strength, sufficient formability, high corrosion resistance, passivity capacity and cost-effectiveness in the production of complex implant shapes are main advantages of titanium [11]. Ti6Al4V and cpTi are the most commonly used titanium powders for medical implants [16]. General properties of Ti6Al4V and cpTi are listed in Table 2.1.

Table 2.1 : General properties of cpTi and Ti6Al4V stated in ASTM F136 and ASTM F67.

Properties	cpTi				Alloy
	Grade 1	Grade 2	Grade 3	Grade 4	Ti6Al4V
Density(g/cm ³)	4.5	4.5	4.5	4.5	4.5
Chemical composition (O,F,N are distinctive)					
Nitrogen	0.03	0.03	0.05	0.05	0.05
Carbon	0.10	0.10	0.10	0.10	0.08
Hydrogen	0.015	0.015	0.015	0.015	0.0125
Iron	0.20	0.30	0.30	0.50	0.25
Oxygen (affects strength and ductility)	0.18	0.25	0.35	0.40	0.13
Tensile strength(Mpa)	240	345	450	550	860
Yield strength(%0.2offset) (Mpa)	170	275	380	485	795
Elongation(%)	24	20	18	15	10
Reduction of area(%)	30	30	30	25	25

In this study, Grade 2 cpTi will be used for the production of samples. In the real application, cpTi is used for the production of cranio-maxillofacial implants as can be seen in Figure 2.10.

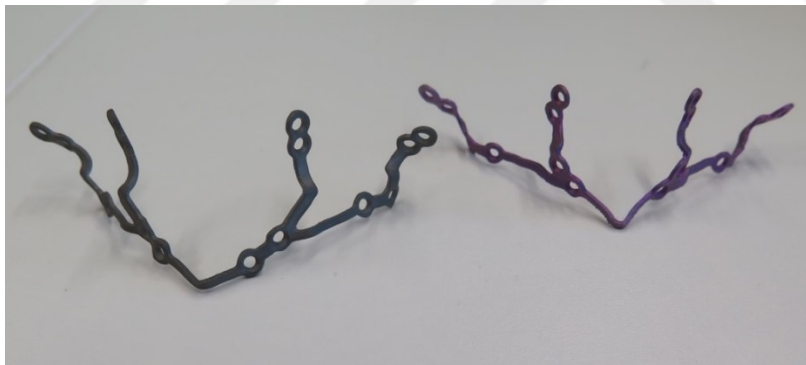


Figure 2.10 : SLM processed cpTi CMF implants.

In order to be able to produce medical implants with SLM, besides chemical or intrinsic properties discussed above, Ti parts should provide certain mechanical properties. Therefore high accuracy and appropriate surface roughness must be ensured by the process [39].

2.3.1 Roughness and implants

Osseointegration which is defined as a direct structural and functional connection between living bone and the surface of a load-carrying implant is affected by roughness [40]. It is stated that the ideal surface roughness (Ra) value for

osseointegration enhancement is between 0.6 and 2.1 μm [6]. Apart from its biological effect on osseointegration, surface roughness has an effect on failure of the implants. Even positive and negative effects of roughness are contradictory; it is a known fact that roughness affects mechanical properties [4, 6]. Hurtado et al. examine the effect of roughness on aluminum sheets and observed that ductility decreases with increased roughness and shear strength is positively affected by roughness [8]. Even though there is no significant effect on static mechanical properties, because of possible notching effect on the surface, fatigue life of the material is affected by surface roughness [7]. Shemtov-Yona et al. stated that when the fractured implants are examined, in addition to main crack, there are secondary cracks that occur as a result of surface roughness [6]. On the other hand, Guilherme et al stated that for cast cpTi with the roughness value of $R_a=0.3\mu\text{m}$ there is no effect on fatigue life [41].

2.4 Definition of Roughness

Amongst many definitions, it can be denoted that roughness is dimensional irregularity of a surface, its deviations from a mathematical flat [42, 43]. It is characterized by hills and valleys of varying amplitudes and spacings [43]. Roughness of a surface can be defined by a touch or appearance of the surface, however the result will be subjective. Quantifying it and getting objective information is termed as a roughness measurement [44]. Roughness is a part of the surface texture. Surface texture is defined by Bhushan as “repetitive or random deviation from the nominal surface that forms the 3D topography of the surface”. It includes not only roughness, but also waviness, lay and flaws as can be seen in Figure 2.11 [43]. Surface texture is not only a performance criterion as in shape, look or mechanical function, but also the fingerprint of the manufacture. Therefore characterization of surface texture will provide controlled quality with prediction and optimization for processes like SLM [44].

Roughness as a fundamental part of the surface texture affects performance and it is directly related to production process as mentioned before.

For the purpose of roughness characterization, the first step is roughness measurement [38]. To be able to define the surface features, irregularities on the surface that can be in many different scales are specified. Different scales mean that

when a surface is specified as “rough”, that doesn’t imply a certain frequency of prominences [45]. For instance, roughness of an asphalt road and roughness of sandpaper or simply a brush are not the same. According to the spacing of irregularities, complex surface texture can contain not only roughness, but also waviness, lay and form in combination (Figure 2.12). As a consequence, distinguishing irregularities is one of the main difficulties about roughness characterization [44].

Irregularities exist on surface are defined as below [43,44];

Roughness: irregularities occurred inherently during production.

Waviness: irregularities of longer wavelengths that overlaps with roughness and it is also called macroroughness.

Form: it is defined as general shape of the surface, prevailed shape when roughness and waviness are neglected.

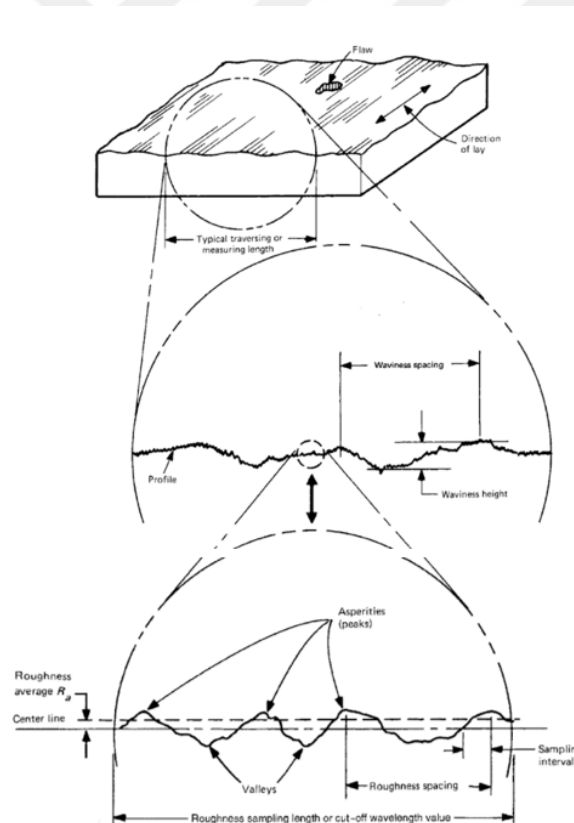


Figure 2.11 : Pictorial display of surface texture [43].

However, there is not an exact point that can be defined as a transition from waviness to roughness or form to waviness. Generally the size of the workpiece defines it [44].

Finally, lay is also a part of surface texture and important for roughness measurement. The lay is defined as the principal direction of predominant surface pattern, called surface pattern generally [43]. The angle between the direction of lay and measured trace is also effective on measurement [44].

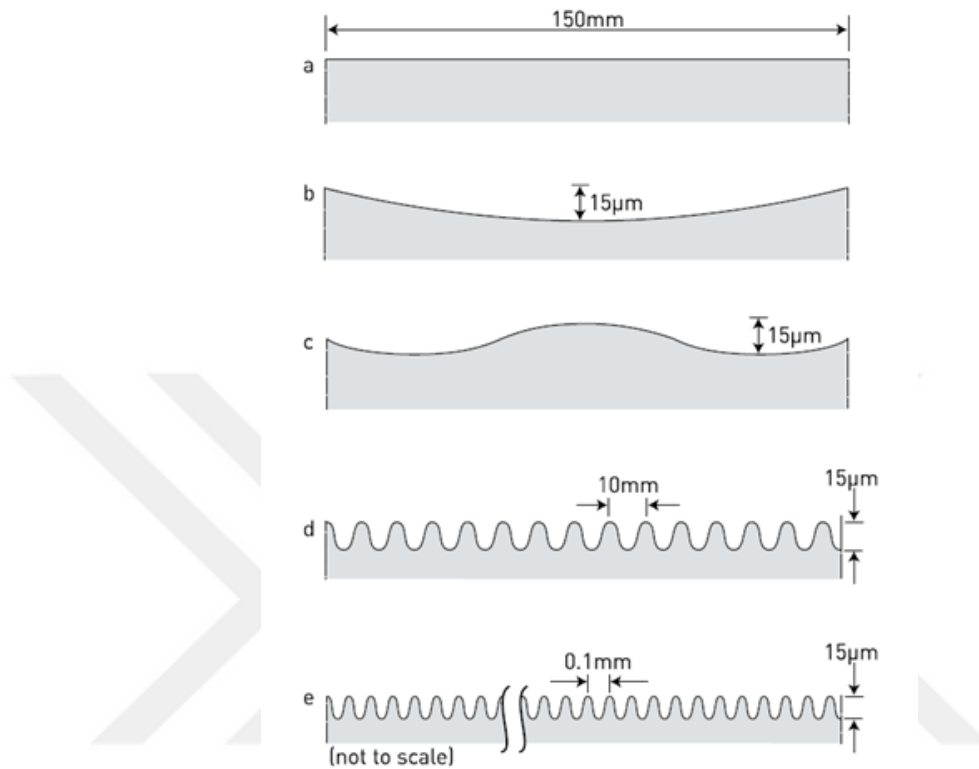


Figure 2.12 : Surfaces having the same irregularities but different spacings. a) smooth, b) smooth but slightly curved, c,d) wavy, e) flat but rough [44].

2.5 Principles of Roughness Measurement

Roughness is not a property that can be directly measured and defined in one value like mass or volume. In order to measure roughness, texture-related information should be extracted from the complex information obtained from measurement and should be turned into useful numbers called roughness parameters [1].

In order to overcome the difficulty about complex nature of texture, many different parameters have been defined for surface characterization [44].

To be able to calculate the correct parameters, a measurement technique should be chosen carefully. This indicates consideration of four; the scales of features to be characterized, the size and shape characteristics that are important for the function of the part, the surface features that give information about manufacturing details and the need for comparative or absolute measurement [1, 45].

And finally, range and resolution is also a phrase that should be explained since it is the limitation for roughness measurement techniques or instruments. It can be defined as the capability of the technique or instrument for measurement. Limitations of range and resolution can be specified as; the largest and smallest differences of height which will be resolved and the longest and shortest surface wavelengths that will be managed. Stedman has defined the envelope to show range and resolution in which instrument or technique is capable of measuring roughness [46]. As can be seen in Figure 2.13, there are some empty regions (some methods are omitted for simplicity). The emptiness like in upper left of diagram means that for measuring large amplitudes at short wavelengths (like a hairbrush), there is no available technique for now [45].

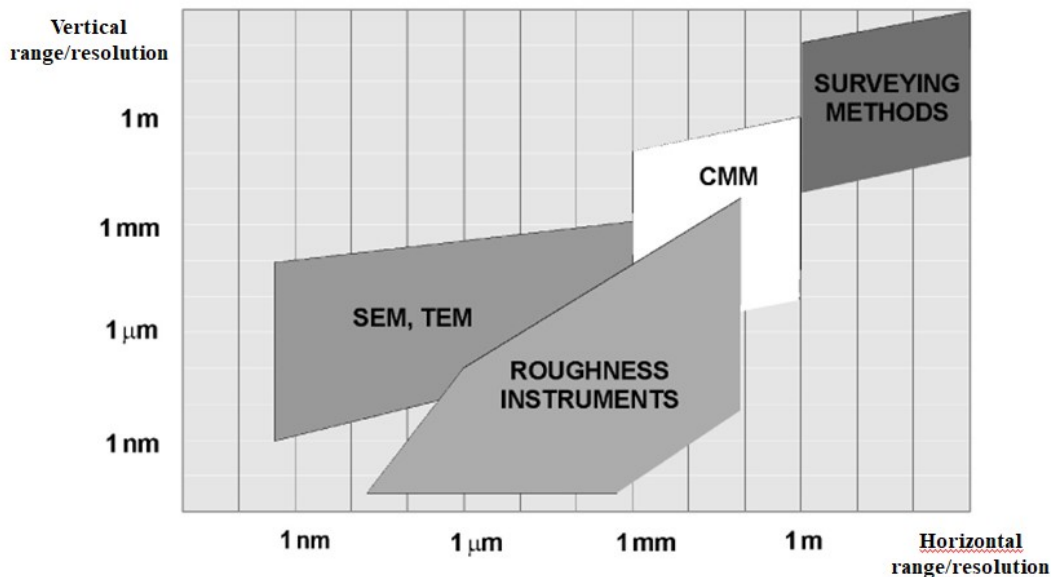


Figure 2.13 : Stedman diagram showing vertical and horizontal resolutions for different roughness measurement methods [46].

Measurement methods can roughly be grouped as; sectional (profiling, 2D) and areal-topography (3D) methods [47].

2.5.1 Profiling (2D) method

The surface roughness is measured along an arbitrary line. Profiling method produces a two-dimensional graph or profile of the surface irregularities as measurement data, which may be represented mathematically as a height function $z(x)$ as illustrated in Figure 2.14 [47].

Some examples of instruments for profiling can be listed as contact stylus scanning (most commonly), early versions of the phase-shifting interferometer, and the optical differential profiler [47, 24].

2.5.2 Areal (3D) method

The surface roughness is measured over an arbitrary rectangular range. Areal method produces a topographical image of the surface, which may be represented mathematically as a height function $z(x, y)$ of two independent variables (x, y) as illustrated in Figure 2.14 [24].

Some examples of instruments (ISO 25178-6) for areal method are contact stylus scanning, phase-shifting interferometric microscopy, coherence scanning interferometry, confocal microscopy, confocal chromatic microscope, focus variation microscopy, optical differential profiler, digital holography microscopy, point autofocus profiling, angle-resolved scanning electron microscopy (SEM), SEM stereoscopy, scanning tunneling microscopy, and atomic force microscopy [1].

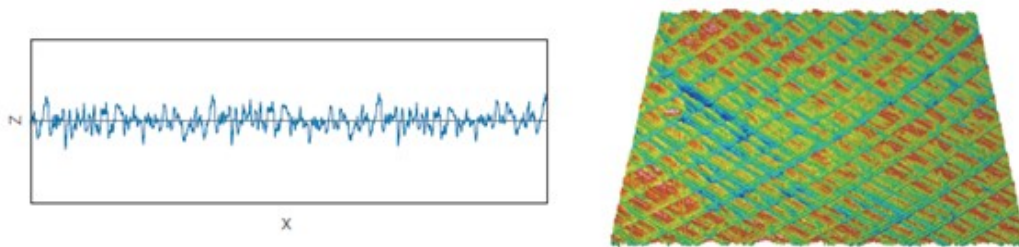


Figure 2.14 : Example of measurement using profile method (left) and Areal method (right) [24].

2.5.3 Profiling vs. areal

The most commonly used method to measure roughness is 2D method for more than half a century [48]. Generally they are advantageous because the data that is obtained directly tracing the surface is considered reliable. Also, properties like low cost, low requirement for training and comfortable nature for inspectors and machinists are other merits for using a profiling method.

Further, in standards, application of profiling is well established (ISO 3274, ISO 4287, ISO 4288, ASME B46.1)[1].

However, profiling might be destructive for soft surfaces and the data may not reflect the whole structure since it is from a single section [47]. Besides, if surface doesn't

have a simple topography with a dominant lay, the capabilities of these methods are limited.

This is the point where areal topography becomes attractive. Specifically, focus variation microscopy and confocal microscopy are ideal optical technologies for measuring the roughness of metal AM parts [1, 13]. There are many non-contact areal methods which can measure without damaging the surface and since it is areal, data includes characteristics of a large surface area. However, for areal measurements, since there are many different principles, on the contrary of profiling, measurement requirements are variable and should be determined by users [47].

2.5.4 Terminology

Evaluation length: The area from which data is obtained. For areal methods, it is a three dimensional area that corresponds to the instrument field of view, or a two dimensional profile that corresponds to the length in the direction of x-axis [47]. It is specified in the literature and standards as in ISO4288-1996 that evaluation length equals to five sampling lengths.

Sampling length (cut-off wavelength): The length that is used for determining the surface characteristics of profile. Any surface irregularities spaced further apart than the sampling length are considered as waviness. Sampling length is also known as cut-off length and considered as representative [47].

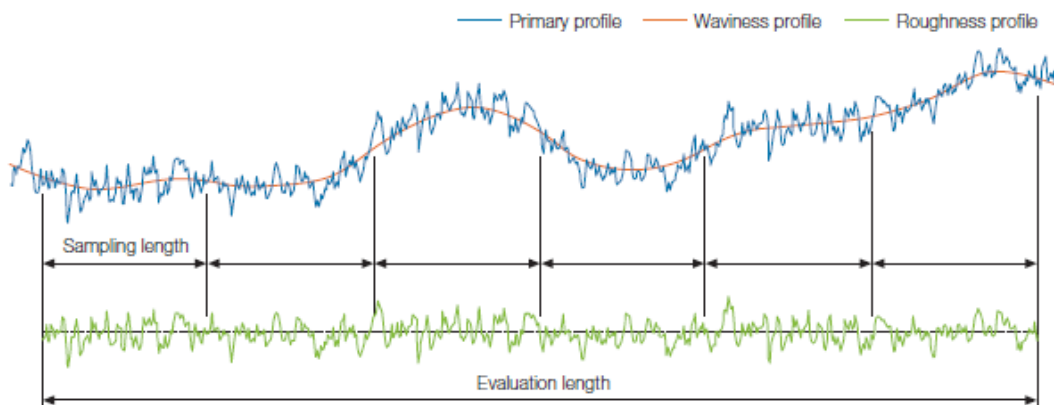


Figure 2.15 : Schematic definition of sampling length and evaluation length on profile curves [47].

Cut-off filter: Determines the wavelength at which the surface structure is differentiated between roughness and waviness data, it acts like a threshold. Proper

selection of the correct filter as cutoff in software is critical for measurement accuracy (λ_c) [47].

Primary profile: The blue profile curve in Figure 2.15 which is obtained by applying long-range filter (λ_s) to eliminate short wavelength portion. Primary profile parameters (P) are calculated from this curve [47].

Waviness profile: The orange profile curve in Figure 2.15 which is obtained by applying λ_f/λ_c filters shown in Figure 2.16. Waviness parameters (W) are calculated from this curve [47].

Roughness profile: The green profile curve in Figure 2.15 which is obtained by applying high-range filter (λ_c) to eliminate primary curve and long wavelength portion. Roughness parameters (R) are calculated from this curve [47].

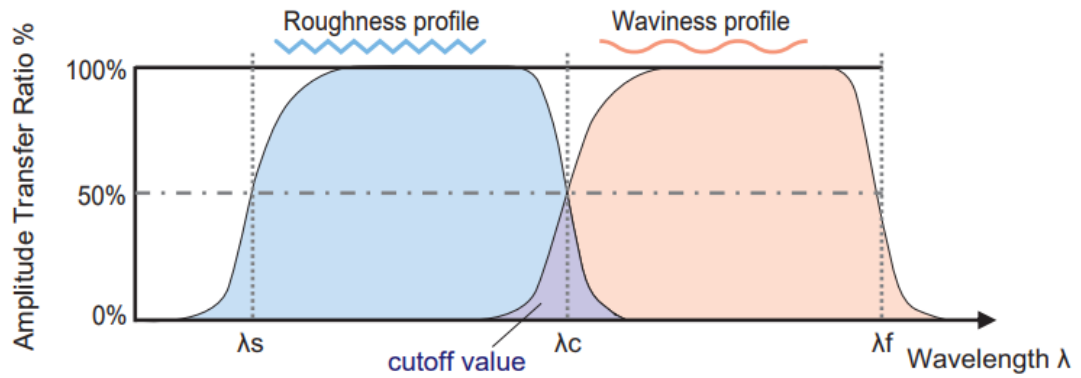


Figure 2.16 : Representation of cut-off filter [47].

2.5.5 Parameters

Surface roughness parameters express the complex geometric characteristics of the surface. They are classified into 3 groups as primary curve parameters (P), roughness curve parameters (R), and waviness curve parameters (W) (ISO 4287:1997). Also, in the recent years, areal ranges parameters (S) have been developed.

When they are grouped according to the dimensions, 2D parameters are P, R, W and 3D parameters are S and V(volumetric) [47].

Totally there are more than 60 defined parameters as can be seen in Table 2.2 [47].

The ones commonly used in present work to compare different methods are described in detail below and shown in Figure 2.17.

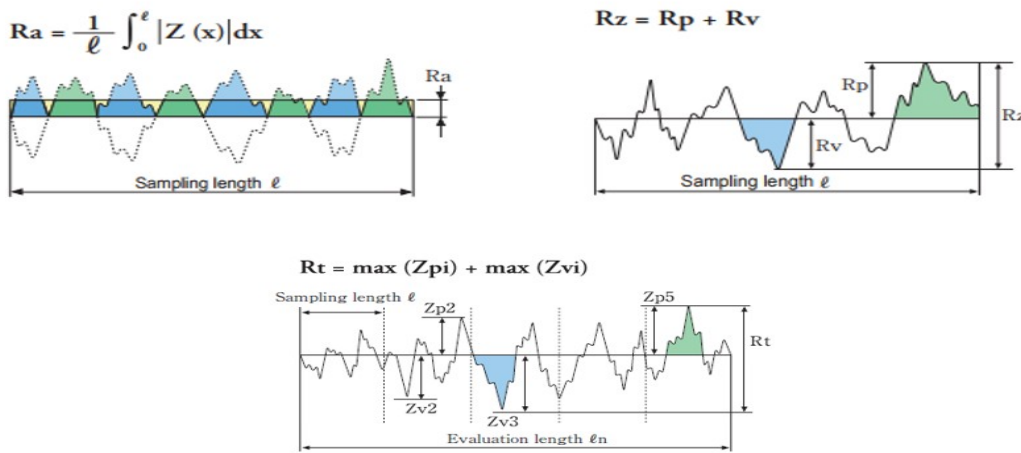
Table 2.2 : Surface roughness parameters [47].

Profile Type	Parameters
Primary Profile	Pp, Pv, Pz, Pc, Pt, Pa, Pq, Psk, Pku, PSm, PΔq, Pmr(c), Pδc, Pmr
Roughness Profile	Rp, Rv, Rz, Rc, Rt, Ra, Rq, Rsk, Rku, RSm, RΔq, Rmr(c), Rδc, Rmr, RzJIS
Waviness Profile	Wp, Wv, Wz, Wc, Wt, Wa, Wq, Wsk, Wku, WSm, WΔq, Wmr(c), Wδc, Wmr Bearing Area Curve

Arithmetic average height (Ra): “It is the most universally used roughness parameter and defines arithmetical mean of the absolute values of Z(x) in a sampling length”[47].

Maximum height (Rz): “It represents the sum of the maximum peak height Zp and the maximum valley depth Zv of a profile within the reference length “[47].

Total height (Rt): “The sum of the maximum peak height Zp and the maximum valley depth Zv of a profile within the evaluation length” [47].

**Figure 2.17 : Schematic presentation of Ra, Rz and Rt [47].**

2.6 Roughness Measurement Instruments

There are several measurement techniques available for roughness measurement varying with contact properties and scales of evaluated features. The first contact type measurement technique was developed in 1933 and non-contact type was found after 50 years. After 1985, measuring roughness even on the atomic scale was achieved [43].

Firstly, as choosing the instrument, deciding whether or not there will be a physical contact with the surface is a must. Also, it should be decided whether it will be an areal or profiling measurement and most importantly, what horizontal and vertical range and resolution should be [45].

2.6.1 Contact type

Contact type surface roughness measurement instrumentation which is also referred as tactile surface profilometer (Stylus) is the most common and oldest roughness measurement technique [49,50]. It has been found almost 100 years ago. It can be defined as a conispherical diamond tip that is dragged across the surface while the vertical motion of tip in response to surface topography is being measured [49]. Both areal and profiling methods can be applied by stylus but with typical stylus speed, areal topography can take a long time [44].

Since a tactile profilometer is relatively simple to operate and its application details are defined by standards ISO 4287 and ISO 4288, it is advantageous [11]. However, because of its contact nature, the profilometers tip can damage soft surfaces [44]. Also, as the tip has a radius (it is stated as $2\mu\text{m}$ in standards), it cannot determine smaller valleys than this radius which is a device limitation. However, especially for averaged roughness values and for final profile, it is stated as negligible [44].

Atomic force microscope (AFM) is one of the most common Scanning probe microscopes (SPM) and it is also grouped as contact type instrument [11]. Generally it is used for measurement in smaller scales and very low stylus force is applied compared to a tactile profilometer [44]. A nanometer-scale probe is used to trace the surface [49]. The tip scans a predetermined pattern and interaction signal is recorded.

However, SPM techniques can provide extremely local information as in scale of few nanometers or a few hundred microns [11].

2.6.2 Non-contact type

Non-contact type instruments eliminate the surface damage possibility and they have relatively higher speed than contact-types. Also, they can be rarely more accurate than a stylus measurement. However, there is no contact, they may not notice and measure small valleys or cannot go across the widely changing shapes unlike contact methods.

Non-contact optical instruments are measuring roughness by two different techniques. One group is defined as those that scan a beam or use the profile or areal methods to measure the actual topography of surface. Second group on the other hand, analyses the distribution scattered light and measures a statistical parameter of the surface [49].

For measuring roughness of SLM processed Ti6Al4V, Focus variation microscopy, AFM (counted as non-contact because of low stress), Confocal microscopy (CM) and X-ray computed tomography (XCT) techniques are used as non-contact types [10, 12, 13].

In Table 2.3, the advantages and disadvantages of the most common and available instruments are listed.

Table 2.3 : Pros and cons of available roughness characterization instruments.

Method	Instrument	Pros	Cons
Contact	Tactile Profilometer (Stylus)	- Widely used, - Standardized (ISO, DIN), 2D profiles with scan range of mm or cm feasible - High accuracy	- Limited lateral resolution, - Surface deformation, - Time consuming for 3D images
Contact	Scanning Probe Microscopy (STM, AFM)	Highest resolution	- Limited z range - Distortion of surface in small scale - Only small areas can be measured - High cost
Non-contact	Confocal Microscopy	- High accuracy - Vertical resolution: 3nm - Lateral resolution: 1μm	Signal decreases and becomes unreliable for high surface slopes
Non-contact	Focus Variation Microscopy	Able to measure high sloped surfaces	- vertical resolution: 100nm - lateral resolution: several pixels
Non-contact	Scanning Electron Microscopy	- High resolution, - High depth of focus, - Morphological information	No quantitative topographical info
Non-contact	Optical microscopy	- Easy, fast - Cheap - MATLAB cross section	- Line profile can be extracted but - Non-guarantee z resolution
Non-contact	Interference Microscopy	- Fast (3D images, minutes) - Resolution: vertical 1nm, lateral 0.2μm	High lateral resolution for only small area
Non-contact	XCT	Data can be extracted from internal surfaces	Small ROI

Kohles et al. compared interferometer and stylus profilometer as 2 commonly used roughness measurement methods for implants and proved that accuracy and precision is directly related to measuring method. They measured roughness from SEM images by drawing contours on cross section view and creating profile and concluded that interferometer is the most accurate one on determining SEM measured Ra values and Stylus (tactile profilometer) is the most precise and repeatable one [50].

Amongst the available methods, instruments for the present work are AFM, tactile profilometer, SEM and CM. Considering the easiness, low cost and relatively high speed of a tactile profilometer when compared the others; it is chosen to be used in the thesis project. Also, in order to have comparable results of a more common method to measure roughness of SLM produced Ti parts, CM will be used. Finally, SEM is used to get qualitative information about roughness and “tilting” will be applied to get some quantitative information.

2.7 Conclusion

To conclude, literature has shown that position and orientation have an effect on surface roughness concurrently post processing steps have also shown to have an effect. Roughness is affecting mechanical behavior and there is a need for further investigation about the severity of this effect.

For roughness measurement methods, tactile profilometer is one of the most traditional and trustable, cost and time effective contact type method. CM is commonly used and accurate non-contact type for roughness determination. Finally, SEM is highly preferred qualitative method because of its high resolution and with the “tilting” method, getting quantitative information would be very interesting. Therefore these three methods are selected to be compared and used in order to be able to assess surface roughness.

After roughness method comparison, all parameters but position and orientation will be fixed considering the highest density of the parts. Built parts' roughness will be measured with one method chosen as the most suitable for this application. Post processing will be applied and roughness measurement will be completed to see its

effect on final roughness and mechanical tests (tensile and fatigue testing) will be applied to see the variation of mechanical properties.



3. MATERIAL AND METHODS

3.1 Material: Commercially Pure Titanium Powder

Grade 2 cpTi powder that has the composition stated in ASTM F67 given in Table 3.1 is used. The particle size distribution is specified as 15-45 μm normally distributed in spherical morphology.

Table 3.1 : Chemical composition of Grade 2 cpTi that is used in this study.

Element	Chemical Composition, % (mass/mass)
Nitrogen, max	0.03
Carbon, max	0.08
Hydrogen, max	0.015
Iron, max	0.30
Oxygen, max	0.25
Titanium	balance

3.2 Selective Laser Melting Machine

All samples for this study are prepared in Magics Software (Materialise NV, Haasrode, Belgium) and are built in an M2 cusing Dual Laser System (Concept Laser, General Electric, Boston, Massachusetts, US) (Figure 3.1). Materialise is one of the partners of this thesis work, therefore printing will be completed within Materialise and some process conditions will be determined according to the company's specifications.

The predetermined production parameters considering maximum density of the parts are used. Also, in order to be able to assure uniformity and comparability across the build plate, only one of the two lasers shown with the red in Figure 3.2 is activated during production. From the right side of the chamber, Argon flow is released to remove evaporated particles and clean the air from undesired particles. A recoater

also moves from right to left to spread a new powder layer uniformly over the build plate.



Figure 3.1 : Selective laser melting machine (M2 cusing GE Concept Laser).

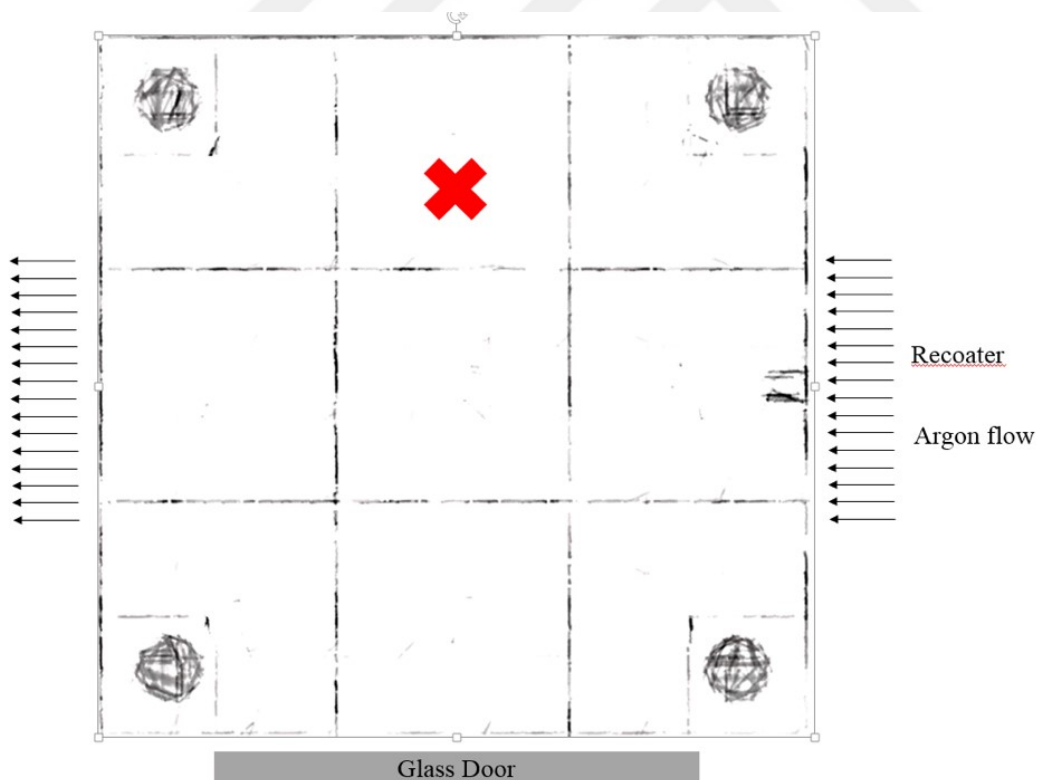


Figure 3.2 : Top view of build plate that the parts are built on and location of laser indicated via a red cross. Ar flow and recoater move from right to left of the build chamber.

3.3 Experimental Outline

The experimental outline is shown schematically in Figure 3.3.

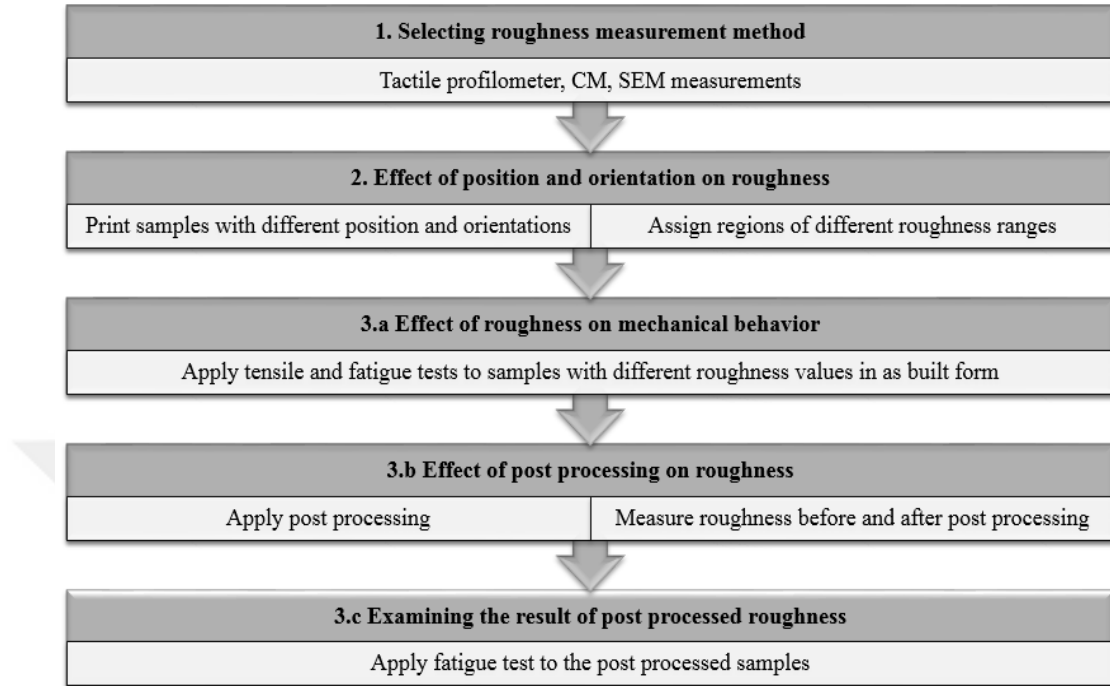


Figure 3.3 : Schematic experimental outline of the thesis work to investigate the effect of roughness on mechanical behavior on cpTi implants produced by SLM.; 1. Selecting roughness measurement method, 2. Determining the effect of position and orientation on roughness and 3. Determining the effect of roughness on mechanical behavior.

Firstly, different measurement methods are compared on different types of samples to determine the measurement method that will be used for all samples of this thesis work.

Secondly, the effect of position and orientation of the parts on the build plate is investigated. In order to do so, all parts are printed in a single batch. Roughness measurements of these parts is done with the selected method and the roughness range across the build plate is determined.

Finally, another build is designed to characterize the mechanical behavior of samples with different surface roughness values.

As mentioned in the literature review, post-processing has an effect on controlled roughness for implants, therefore anodization will be applied on the produced parts to differentiate their roughness values prior to mechanical testing.

Varying roughness values of different samples will be measured with the selected method. Then, the samples in their as-built (AB) and post-processed (PP) states will undergo tensile and fatigue testing in order to have information on their mechanical behavior.

3.4 Roughness Measurement

For determining a roughness measurement method suitable for cpTi components produced via SLM, rectangular samples are designed in Magics Software (Materialise NV, Haasrode, Belgium) and printed in the same build within the same region of the build plate. The samples have a thickness, width and length of 1 mm, 12 mm and 70 mm, respectively (Figure 3.4).



Figure 3.4 : Sample design that is used to determine roughness measurement method. Thickness is 1 mm, width is 12 mm and length is 70 mm. 6 of this sample design are produced within the same region (right bottom corner) of the build plate.

Prepared samples are listed with their names and specifications in Table 3.2. Sand blasting (SB) is used to differentiate roughness. In total, there are 6 samples which can be broken down into sub-categories (with 2 samples for each category); as-built (AB), sandblasted for 30 seconds (SB30) and sandblasted for 60 seconds (SB60) respectively.

Table 3.2 : Sample list used for determining roughness measurement method with the specifications. “AB” stands for the samples at as built state whereas “SB30” stands for samples that are sandblasted 30 for seconds and “SB60” stands for the parts that are sandblasted for 60 seconds. The letter “B” on the sample names refer to the “back side” of the sample and “F” refers to the “front side”.

Part ID	F1	B1	F2	B2	F3	B3	F4	B4	F5	B5	F6	B6
Status	AB	AB	AB	AB	SB	SB	SB	SB	SB	SB	SB	SB
Time(sec)					30	30	30	30	60	60	60	60

In Table 3.2, the letter “B” on the sample names refer to the “back side” of the sample and “F” refers to the “front side”.

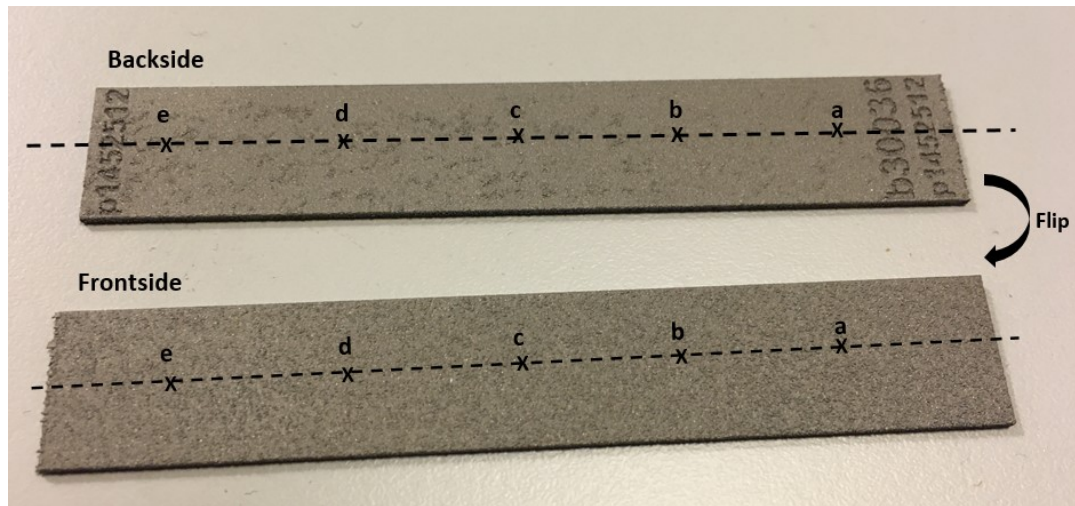


Figure 3.5 : Measuring strategy on the front and back side of the sample.

For consistency, measurements are taken across the 5 interval point shown a-e. all measurements are done on the middle line along the sample and 5 points (a, b, c, d, e) are used to specify areas on the sample as shown in Figure 3.5.

As stated before, roughness measurement method and cut-off length directly rely on the sample that is being measured. Cut-off length determines the range of irregularities that will be considered as roughness. Measurements have been done with different cut-off lengths to find the most suitable cut-off length for these samples while still being possible to apply on due to the sample dimensions. By visual inspection and measurements that are done vertically and horizontally, the surface properties of the samples are specified as non-periodic, so roughness ranges and cutoff lengths listed in the ISO 4288 for non-periodic profiles shown in Table 3.3 are considered.

Table 3.3 : Cut-off (Sampling) lengths and evaluation lengths for specific Ra and Rz ranges stated in ISO 4288 – Geometric Product Specification (GPS)- Surface Texture- Profile method: Rules and procedures for the assessment of surface texture for non-periodic profiles.

Ra μm	Rz μm	Sampling Length mm	Evaluation Length mm
$0,006 < \text{Ra} \leq 0,02$	$0,025 < \text{Rz} \leq 0,1$	0,08	0,4
$0,02 < \text{Ra} \leq 0,1$	$0,1 < \text{Rz} \leq 0,5$	0,25	1,25
$0,1 < \text{Ra} \leq 2$	$0,5 < \text{Rz} \leq 10$	0,8	4
$2 < \text{Ra} \leq 10$	$10 < \text{Rz} \leq 50$	2,5	12,5
$10 < \text{Ra} \leq 80$	$50 < \text{Rz} < 200$	8	40

When the measured Ra value is in the range stated in Table 3.3, the cut-off length that is being used is set and specified as the correct cut-off for the method and the sample. Due to the common ability of the three methods to calculate Ra and since Ra is the most conventional roughness parameter, Ra values are used for comparing between methods.

3.4.1 Confocal microscopy

As the first measurement method, multi-pinhole disc scanning confocal microscopy (CM) (μ surf explorer, Nanofocus, Germany) is used. For this technique samples are provided to Falex Tribology (Leuven, Belgium) for measurement. CM is simply collecting 3D data in x-y-z directions and turns them into roughness parameters of the surface and since it is collecting data and directly measuring, it can also create 2D profiles and calculate 2D roughness parameters.

For every sample, cut-off lengths of 0.25 mm, 2.5 mm and 8 mm are used. The 3D profiles of the areas measured (1.5 mm x 1.5 mm for 0.25 mm cut-off and 2.95 mm x 40 mm for 2.5 and 8 mm cut-offs) are shown in Figure 3.6.

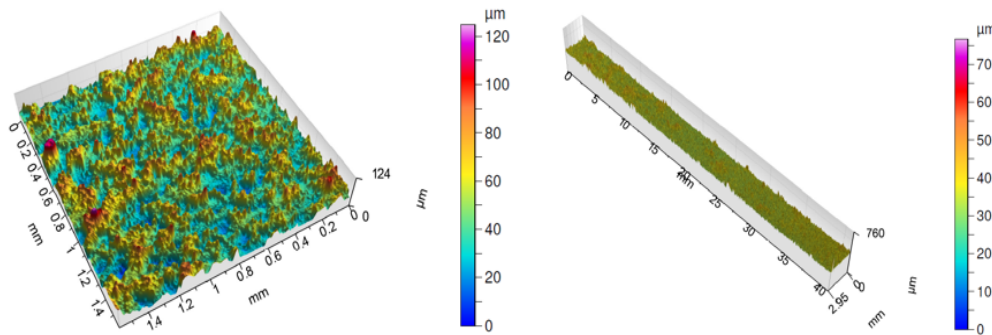


Figure 3.6 : 3D surface profiles that are acquired by CM with the cut-off lengths of a) 0.25 mm and b) 8 mm cut-off lengths (belongs to sample B1 at point 'a').

As shown in Table 3.3, for a cut-off length of 0.25 mm, an evaluation length of 1.25 mm is needed, therefore 5 measurements at the points previously defined (see Figure 3.5) are done for each side of the samples (5 measurements x 6 samples x 2 sides = 60 measurements for 0.25 mm cut-off length). On the other hand, an 8 mm cut-off length has an evaluation length of 40 mm and due to the samples dimensions x = 70 mm, only one measurement along the middle line is completed (6 samples x 2 sides = 12 measurements).

Roughness parameters are calculated and recorded by Mountains Software (Digital Surf, France). Software is using the images to draw lines on it and extract the surface profiles along the lines. To be able to have the data for all area, software is set to draw maximum amount of profile lines on the area being examined. Then, profile curves as shown in Figure 3.7 are created and roughness parameters for every line profile are averaged to calculate roughness parameters of the samples.

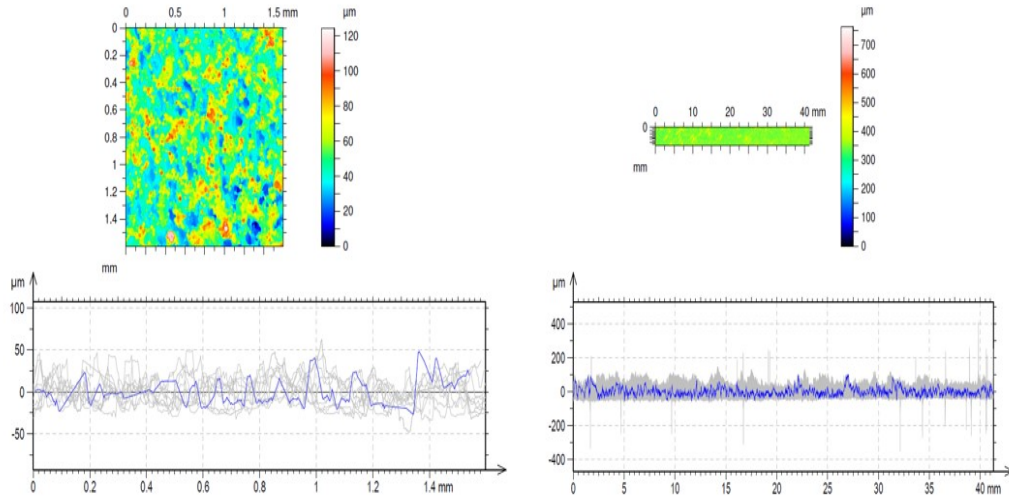


Figure 3.7 : Profile curves created by Mountains Software to calculate roughness parameters of the samples. The squared area is acquired with the cut-off length of 0.25 mm and rectangular area is acquired with the cut-off length of 8 mm.

3.4.2 Tactile profilometer

A tactile profilometer (Stylus) (MarSurf PS1, Mahr, Germany) with a tip size of 2 μm in Figure 3.8 is used. While stylus is traversing the surface, the movement in z direction is recorded and roughness parameters are displayed. The average surface roughness (R_a ; μm), the arithmetic mean height of the surface profile (R_z ; μm) and the total height of profile (R_t ; μm) are measured. The tactile profilometer has 3 settings in terms of cut-off length options; for every sample, cut-off lengths of 0.25 mm, 0.8 mm and 2.5 mm are used. Larger cut-off lengths are not employed due to the available settings of the stylus.

For 0.25 mm and 0.8 mm cut-off lengths, 5 measurements are completed along the middle line of the sample for each side and average R_a values are calculated (5 measurements x 6 samples x 2 sides = 60 measurements for each cut-off length).



Figure 3.8 : MarSurf Stylus Instrument PS1 with the tip size of 2 μm .

On the other hand, for a 2.5 mm cut-off length which is the maximum cut-off length available for Stylus, 3 measurements are completed along the middle line. First measurement is carried out on the line from point a to point c, second is from point b to point d and final measurement is from point c to point e (3 measurements x 6 samples x 2 sides = 36 measurements for 2.5 mm cut-off length) (see Figure 3.5).

3.4.3 SEM

SEM images as qualitative data are taken with Nova NanoSEM 450 (FEI) (Thermofisher Scientific, Japan). SEM images are then converted into quantitative data (3D MeXSoftware, Alicona, Austria). In order to have quantitative data with SEM, 2D images are used to create 3D data by image processing. By combining two SEM images taken with 0° and 30° tilted (Figure 3.9) and applying image processing, form on the surface has been removed and roughness profile is obtained.

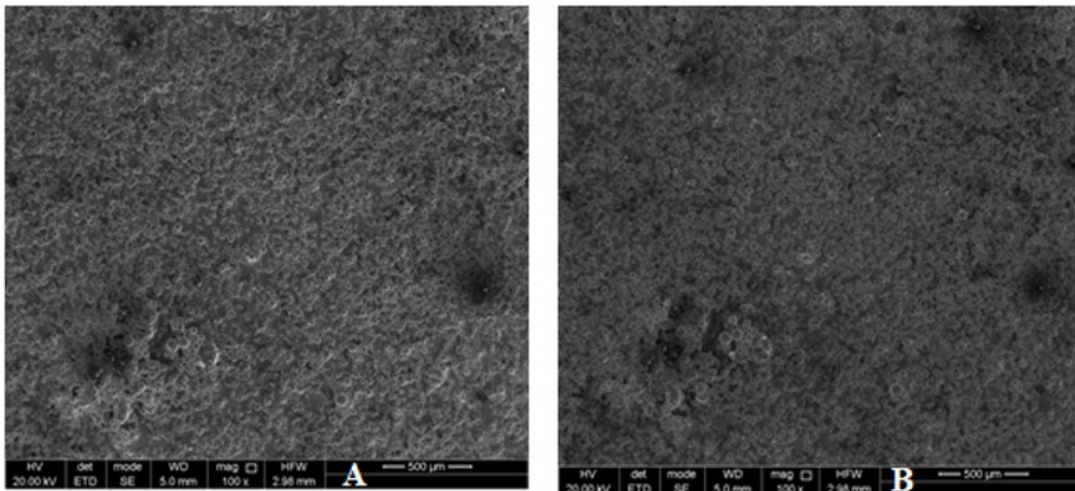


Figure 3.9 : SEM images with 100x magnification of sample B1 at point 'c' A) 0° B) 30° tilted

Results of different image processing steps are shown in Figure 3.10.

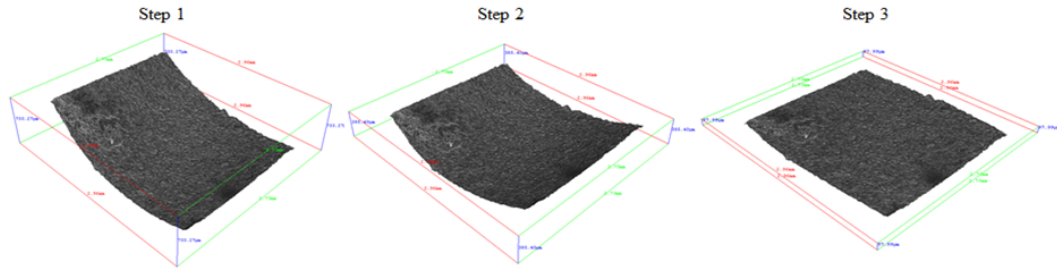


Figure 3.10 : Results of different image processing steps. Step 1 is the combination of tilted and non-tilted image. Step 2 means removal of tilt form and Step 3 is the results of cylindrical form removal and where the final surface roughness measurements will be calculated from.

3.5 Mechanical Characterization

3.5.1 Mechanical testing

Prior to mechanical testings, surface roughness measurements with tactile profilometer (MarSurf PS1, Mahr, Germany), plus thickness with micrometer and width measurement with a digital caliper (Mitutoyo, Japan) of the samples is completed.

To be able to apply mechanical tests and measure roughness easily, sample shape is determined as dog-bone tensile bar. This is chosen to allow identical samples to be used for both fatigue and tensile testing while limiting any effects which sample dimensions might have on surface roughness properties. The dimensions are calculated according to the standards ISO 6892 (Metallic Materials - Tensile Testing), ISO 1099 (Metallic Materials - Fatigue Testing) and ASTM E466 (Fatigue Testing). Dimensions are specified to ensure both tensile and fatigue test specimen conditions are met as shown in Figure 3.11.

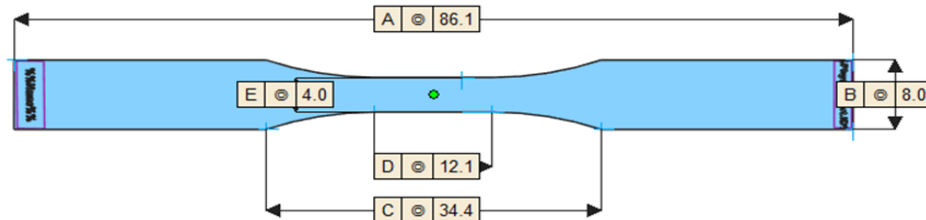


Figure 3.11 : Tensile bar design for mechanical testing. Dimensions are specified according to ISO 6892, ISO 1099 and ASTM E466 to ensure both tensile and fatigue test specimen conditions are met. (total length: 86.1 mm, width at grips: 8 mm, thickness: 0.8 mm, R=32 mm, total grip length: 50 mm).

3.5.1.1 Tensile test

Tensile tests are carried out using a Zwick/Roell Proline Z050 (Zwick, Germany) model testing machine with 2.5 kN load cell, wedge grip and automatic 'makroXtens' extensometer (see Figure 3.12).

Test conditions can be listed as; pre-load: 5 MPa, gage length: 12 mm, speed in elastic region: 0,006 mm/mm/min, speed after yield: 0,07 mm/mm/min, end of test: failure.

For every group of sample (5 positions are determined to build on and create 5 different types of samples), 5 replicates are prepared and tested (Table 3.4). For each sample the roughness range and average of the yield strengths obtained from 5 measurements is used to calculate the initial stress values used for that roughness range during fatigue testing.



Figure 3.12 : Tensile test set-up.

Table 3.4 : Specifications and number of samples used for tensile tests. 5 replicates are used for every positions. ‘AB’ refers to the as built samples, ‘MPP’ refers to the post-processed samples with standard Materialise post-process steps and ‘SB’ refers to the sandblasted samples.

Number of different positions	Status	Applied test	Number of samples
5	AB	Tensile	25
1	MPP	Tensile	5
1	SB	Tensile	5

3.5.1.2 Fatigue test

The ElectroPulsTM E300 (Instron, Massachusetts, US) dynamic test equipment (see Figure 3.13) and Wavematrix Software (Instron, Massachusetts, US) are used to get fatigue data.

Load controlled, tension-tension fatigue tests are applied. An R-ratio is determined as 0.1 and frequency is set to 60Hz.

80% of average yield stress calculated by tensile tests is used as fatigue test input. Stress levels are determined as 180 MPa, 225 MPa, 292 MPa, 360 MPa and 450 MPa and these same 5 levels are used for every sample to assure comparability. 3 samples are used for each stress level (except for the min and max stress levels where 2 samples are used). 1 million cycles is determined as a runout since it is the lifetime defined for CMF implants.

AB samples from minimum and maximum roughness ranges and PP samples from the max region are selected to be tested to see the effect of surface roughness on fatigue behavior. Also, Sample specifications and amounts are given in Table 3.5.

Table 3.5 : Specifications and number of samples used for fatigue tests. 3 replicates are used for 4 stress levels lower than 450 MPa. ‘AB’ refers to the as built samples, ‘MPP’ refers to the post-processed samples with standard Materialise post-process steps and ‘SB’ refers to the sand-blasted samples.

Specification	Status	Applied test	# of samples
Max	AB	Fatigue	13
Min	AB	Fatigue	13
Max	MPP	Fatigue	14
Max	SB	Fatigue	13



Figure 3.13 : Fatigue test set-up (Instron). Load controlled, tension-tension fatigue tests are applied with $R = 0.1$ and runout = 1 million cycles.

3.5.2 Microstructure

For microstructure analysis; parts are embedded (Epofix), polished, grinded (Tegramin 20, Struers, US) and etched (Kroll's Reagent). As the microscope, Olympus BX 51 (Tokyo, Japan) is used.

3.6 Post Processing

3.6.1 Heat treatment

Before removing parts from the build plate, stress relief heat treatment is applied to all parts to avoid thermal stresses.

3.6.2 Anodization

As post processing that affects roughness, standard Materialise post-processing (MPP) steps are applied as shown in Figure 3.14.



Figure 3.14 : Materialise post-processing (MPP) applied for cpTi implant applications

4. RESULTS AND DISCUSSION

4.1 Roughness Measurement Method

For comparing different roughness measurement methods, Ra values are used and samples are examined considering front and back sides separately. As stated in material and methods, different cut-off lengths are applied to find the correct cut-off length stated in Table 3.3 for different methods. Preliminary results showed that 0,25 and 0,8 mm cut-off lengths are small and 2,5 and 8 mm cut-off lengths should be applied to find the correct cut-off length with regards to the sample dimensions and three roughness values encountered.

Visual inspection has showed that as the sandblasting (SB) time (which is changed to differentiate roughness) increases, surface looks less rough. Also, there is a clear difference between front and back side of the samples in terms of roughness; Front side has a rougher appearance than the back side (Figure 4.1).



Figure 4.1 : Front side of the sample displaying rougher appearance than the back side.

4.1.1 Confocal microscopy

The measurement results of CM with different cut-off lengths are shown in Figure 4.2. Firstly, CM is able to detect the expected difference caused by different SB time and different sample sides.

Secondly, it can be easily seen that as the cut-off length increases, the Ra values measured for every sample also increases. This is due to the fact that cut-off length determines which irregularities to be considered as waviness or roughness [47]. It serves as the threshold, and irregularities which are longer than the specified cut-off length are considered as waviness so they are eliminated from the surface profile by filtering [51]. Therefore, as the cutoff length increases, longer irregularities are considered as roughness.

Another important point to mention is 2,5 mm and 8 mm cut-off lengths give the same value for sample B6. Sample B6 is mentioned specifically because that is the only sample that the correct range is reached with 2,5 mm and it is possible to apply a longer cut-off than the correct one for this sample. This proves that once the correct cut-off length is used and roughness values are in the range, even if the evaluation length is increased, since the whole of the profile has already been covered, the Ra value does not change.

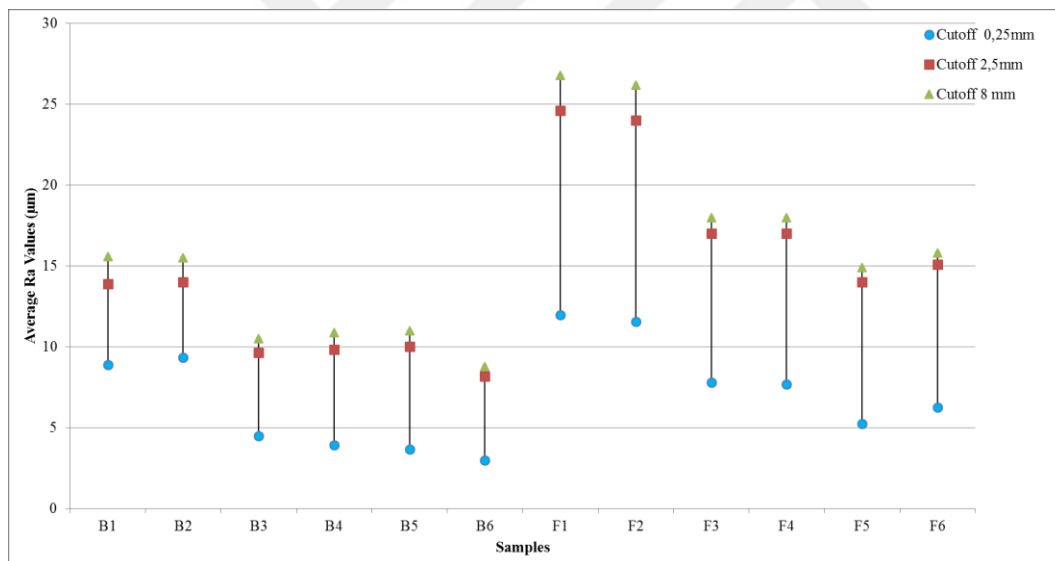


Figure 4.2 : CM Ra results (μm) with different cut-off lengths as in 0,25 mm, 2,5 mm and 8mm. 'B' refers to back side and 'F' refers to front side. Sample 1 and 2 are AB, Sample 3 and 4 are SB for 30 seconds and Sample 5 and 6 are SB for 60 seconds.

As stated in literature, evaluation length and cut-off length are directly proportional, therefore as cut-off increases, evaluation length is also increasing and according to the specimen size and frequency of irregularities, the correct roughness data is collected by applying the right cut-off length. Even if an increase is expected, it can

be stated that the jump between the values for 0,25 mm and 2,5 mm is higher than the difference between the values for 2,5 mm and 8 mm cut-off lengths.

Table 4.1 : CM Ra values (μm) measured for different cut-off lengths of 0,25 mm, 2,5 mm and 8 mm respectively.

ISO 4288 Ra range	Cut off (mm)	B1	B2	B3	B4	B5	B6	F1	F2	F3	F4	F5	F6
0,02-0,1	0,25	8,9	9,3	4,5	3,9	3,6	3,0	12,0	11,6	7,8	7,7	5,2	6,3
2-10	2,5	13,9	14,0	9,6	9,8	10,0	8,2	24,6	24,0	17,0	17,0	14,0	15,1
10-80	8	15,6	15,5	10,5	10,9	11,0	8,7	26,8	26,2	18,0	18,0	14,9	15,8

As can be seen in Table 4.1, for 8 mm cut-off, Ra values vary from 10 to 26 μm (exception: sample 6 gives 8 μm) and ISO 4288 determines the range as 10 to 80 μm . Also, Ra values measured with 2,5 mm cutoff vary from 8 to 24 μm whereas the range is determined as 2 to 10 μm by standards. However, 0,25 mm cut-off values are far more outside of the standardized range. As the cut-off length increases and as getting closer to the correct cut-off length for the sample, more surface features can be seen. Therefore the Ra values are more accurate and the difference between values of different cut-off lengths decreases as getting closer to the correct cut-off.

When all results are compared to ISO 4288, it is concluded that 8 mm cut-off length is the correct cut-off length for the tested samples.

4.1.2 Tactile profilometer

Measurement results of tactile profilometer with different cut-off lengths are shown in Figure 4.3.

It can be concluded from tactile profilometer results that it is also capable of detecting a decrease in Ra as the SB time increases and front sides have higher Ra values than back sides.

Also Ra values are increasing as the cut-off length increases. As shown in Table 4.2, 0,25 mm cut-off length which is stated as the correct cutoff length for Ra values smaller than 0,1 μm , gives Ra values between 2 to 6 μm . Also, 0,8mm cutoff range is defined as 0,1 to 2 μm , whereas the resultant Ra values are between 6 to 14 μm . On the other hand, 2,5 mm cutoff length which represents the range of 2 to 10 μm , gives values between 9 to 19 μm . Even these are not exactly fitting the range of 2,5 mm,

because the maximum applicable cut-off filter for the tactile profilometer used in this thesis is 2.5 mm. 8 mm cut-off length is not applied.

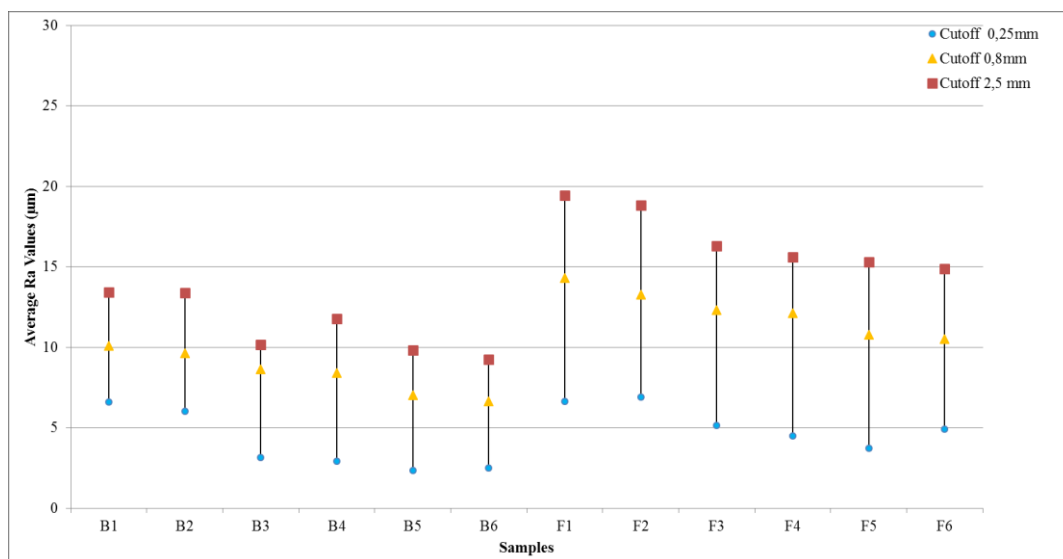


Figure 4.3 : Tactile profilometer Ra results (µm) with different cut-off lengths as in 0,25 mm, 0,8 mm and 2,5 mm.

Table 4.2 : Tactile profilometer Ra values (µm) for different cut-off lengths as in 0,25 mm, 0,8 mm and 2,5 mm.

ISO 4288 Ra range	Cut off (mm)	B1	B2	B3	B4	B5	B6	F1	F2	F3	F4	F5	F6
0,02-0,1	0,25	6,6	6,0	3,2	2,9	2,3	2,5	6,6	6,9	5,1	4,5	3,7	4,9
0,1-2	0,8	10,1	9,6	8,6	8,4	7,0	6,7	14,3	13,3	12,3	12,1	10,8	10,5
10-80	2,5	13,4	13,4	10,2	11,8	9,8	9,2	19,5	18,8	16,3	15,6	15,3	14,9

4.1.3 SEM

Measurement results for SEM are shown in Figure 4.4.

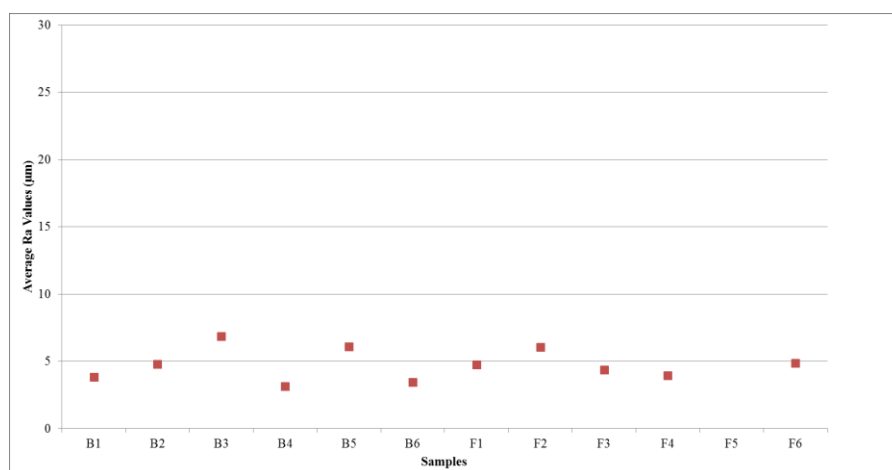


Figure 4.4 : SEM Ra results (µm) for 2,5 mm cut-off length.

The difference between samples that are SB differently and difference between front and back sides are not detected by SEM.

As can be seen from the Table 4.3, values calculated by image processing with SEM gives Ra values between 3 to 6 which all are in the range of 2,5 mm cut-off.

Table 4.3 : SEM Ra values (μm) measured with 2.5 mm cut-off length (measurement error noticed after testing Sample F5).

ISO 4288 Ra range	Cut off (mm)	B1	B2	B3	B4	B5	B6	F1	F2	F3	F4	F5	F6
2-10	2,5	3,9	4,8	6,8	3,1	6,1	3,4	4,7	6,1	4,4	4,0		4,9

4.1.4 Comparison of different methods

For the comparison, only the values that are relevant according to ISO standard will be used. All related three methods' results are shown in Figure 4.5.

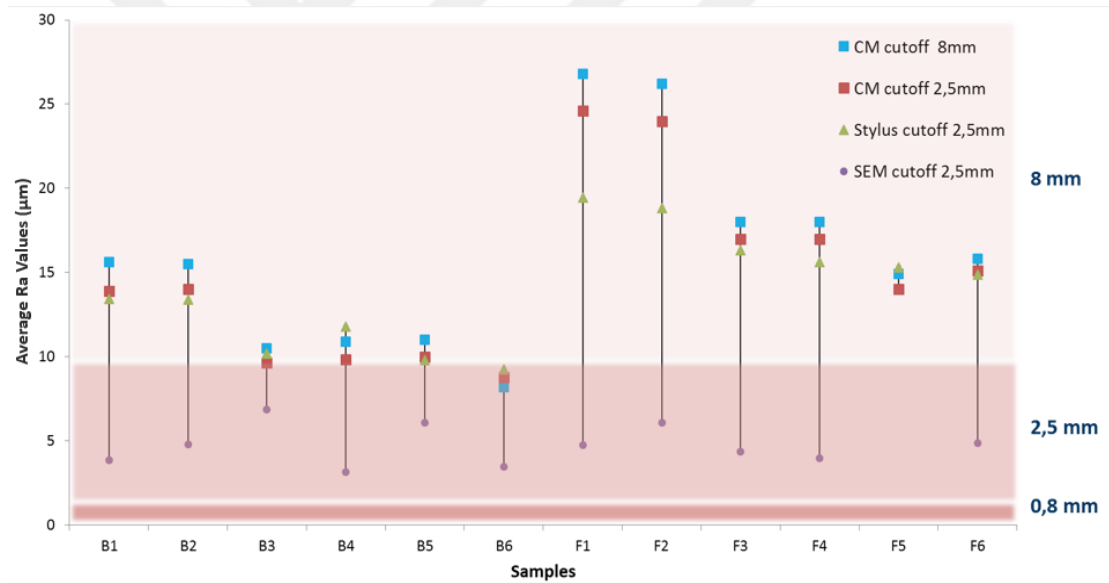


Figure 4.5 : Ra (μm) results of CM with 2.5 mm and 8 mm cut-off lengths, tactile profilometer (Stylus) with 2.5 mm and SEM with 2.5mm cut-off lengths. B` refers to back side and `F` refers to the front. Sample 1 and 2 are AB, Sample 3 and 4 are SB30 seconds and Sample 5 and 6 are SB60.

Firstly, it is observed in CM and tactile profilometer results that they both show the decrease in Ra when SB time increases and they also display the difference between front and back sides. On the other hand, there is no such significant pattern on SEM results that supports the common behavior detected by CM and tactile profilometer.

The observed decrease in surface roughness with increased SB time due to the removal of semi-attached particles that are a natural occurrence of the intrinsic

properties of SLM. In the case of highly rough surfaces with semi-attached particles on, SB causes a decrease in roughness by removing the semi-attached particles that are causing additional irregularity.

The difference between front and back on the other hand was expected regarding the research done by Kleszczynski et al but the reason of this is out of this chapter's scope [5].

Secondly, both CM and tactile profilometer give similar results for the same samples and both methods indicate that suitable cut-off length for these samples is changing between 8 and 2.5 mm depending on the minimum and maximum Ra values measured.

For back sides, the roughness range is between 8-15 μm and both CM with 2,5 mm and tactile profilometer with 2,5 mm cut-off show similar results. Also, CM with 8 mm shows close results to them. However, when roughness increases to 25 μm for front sides (F1 and F2 especially), the difference between 8 and 2,5 mm cut-off lengths is increasing too. This can be explained as an effect of being close to the range of correct cut-off length. As getting closer to the correct cut-off length for the surface being measured, the difference between the measured values decreases. At the point that the correct cut-off length is reached, even if a longer cut-off length is applied, measurement gives the same value.

Another observation is that when roughness increases and exceeds the limits of 2,5 mm cut-off length, the results of CM 2,5 mm and tactile profilometer 2,5 mm are showing variation. The reason of this might be the tip of 2 μm radius. It may not extract information of pits smaller than 2 μm because of the contact type nature of tactile profilometer. CM on the other hand is capable of getting information without physical limitations of traversing.

However, compared to the other two methods, SEM results are significantly lower. SEM with 2,5mm cut-off length, gives much smaller Ra values which are in the range of 2,5 mm that is specified in standards. Despite other cut-off lengths are also tried for SEM, correct range has not been reached with any but 2.5 mm. The reason of SEM giving uncorrelated values with the others can be explained by its relatively small ROI. While we are collecting the information on the area of only 8 mm² (2,98 x 2,74 mm²) for SEM (100x), CM has area of 118 mm² (2,95 x 40 mm²) (for 8 mm

and 2.5mm cut-off) or 36 mm² (2,95 x 12,5 mm²) (for 0.25 mm cut-off) and tactile profilometer has a line length of 12,5 mm. It can be said that because of limited operational range of SEM for these samples with non-periodic high surface roughness, surface features can not be fully covered by SEM. It focuses on a smaller area, even if the 100x magnification is used. That can also explain why bigger cut-off length trials haven't given higher Ra values for SEM. Despite longer discontinuities are allowed to be considered with higher cut-off lengths and the transition point for roughness to waviness/form changes, small ROI can not provide full profile that represents the whole sample with higher roughness. In Figure 4.6, the effect of magnification of SEM can be seen. Both images belong to the same area of the same sample. However, with higher magnification (500x), even if the same cut-off length is applied (blue boxes), different features are get by the measurement. Since SEM has higher magnification than the other two methods, some information can be missed.

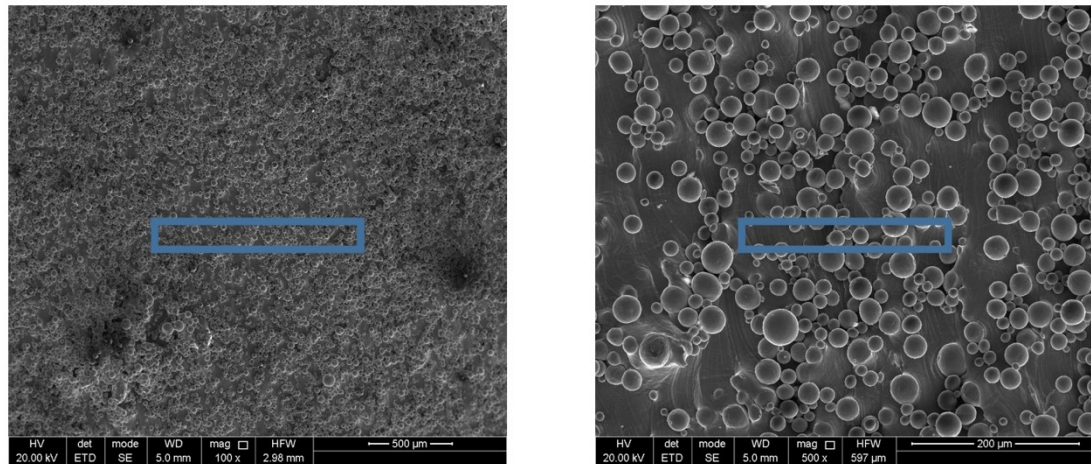


Figure 4.6 : SEM images of the same area with 100x (left) and 500x (right) magnification. Blue boxes represents the same cut-off length and covered features are different.

Also, it can be explained by the tilting technique that is used for SEM. Since the images were taken with a 30 degrees tilt, it may not be enough to observe the peaks fully and can cause measuring the roughness values lower than they are.

Another possible effect for the difference between SEM and other methods are the working principles of these measurement methods. While CM is directly collecting 3D information by taking several 2D images at different z heights, and tactile profilometer is recording the movement of stylus traversing the surface, SEM is

using 2D images turned into 3D data. Therefore, CM and tactile profilometer are more direct methods than SEM.

Additionally, areal methods have some specification stated in ISO 25178, however specifically tactile profilometer (Stylus) has been standardized for determining correct cut-off length and roughness parameters as stated in ISO 4288, so Ra values measured with Stylus can be verified. Also, CM, which is one of the most common surface roughness measurement methods as mentioned in literature review, gives similar results to tactile profilometer. Therefore, the Ra range for the samples being measured can be determined as between 15 and 9 μm for the front and 26 to 14 μm for the back side of the samples as listed in Table 4.4. However, for Ra values higher than 20 μm , it seems that threshold limits are exceeded and tactile profilometer with the limit of 2,5 mm cut-off length may not be sufficient to quantify those roughness levels. For high roughness surfaces, tactile profilometer is giving smaller values than CM because there is no option for 8 mm cut-off length for the tactile profilometer that is being used.

Table 4.4 : Overall CM (8 mm), CM (2,5 mm), Tactile profilometer (Stylus) (2,5 mm) and SEM (2,5 mm) Ra results.

Type-Cutoff	B1	B2	B3	B4	B5	B6	F1	F2	F3	F4	F5	F6
CM-8mm	15,60	15,50	10,50	10,90	11,00	8,18	26,80	26,20	18,00	18,00	14,90	15,80
CM-2.5mm	13,90	14,00	9,64	9,84	10,00	8,18	24,60	24,00	17,00	17,00	14,00	15,10
Stylus-2.5mm	13,44	13,41	10,17	11,77	9,81	9,24	19,45	18,82	16,30	15,61	15,29	14,87
SEM-2.5mm	3,85	4,78	6,84	3,14	6,08	3,44	4,74	6,07	4,35	3,95		4,86

Nevertheless, it can also be concluded that even the values are slightly different, CM and tactile profilometer are giving the same trend so they both can be considered as capable of roughness range characterization across the build plate. Specifically, samples B 3-4-5 and 6 measurements show that tactile profilometer and CM are both capable of measuring and for the roughness values in the 2,5 mm cut-off length range, they both are equally reliable.

Further, since tactile profilometer and CM can both be considered capable, when they are compared for this thesis considering the number of samples to be produced

and measured; tactile profilometer is advantageous in terms of more practical application and cost effectiveness since CM is out-sourced. Therefore, for the next steps, tactile profilometer is used as the roughness measurement method.

4.2 Roughness Range Determination Across Build Chamber

In order to determine the roughness range across the build chamber, 15 positions are determined considering the distance from the laser and symmetry between left and right side of the chamber (see literature review) as you can see in Figure 4.7.

Different positions are named via numbers that are increasing as the distance from the laser increases and left and right side are stated with letters L and R respectively.

For every position, in order to determine the orientation effect, parts with 6 different orientations with different z-rotations are placed. The group of six (shown in Figure 4.8) is then examined orientation by orientation to see the position and orientation effects separately.

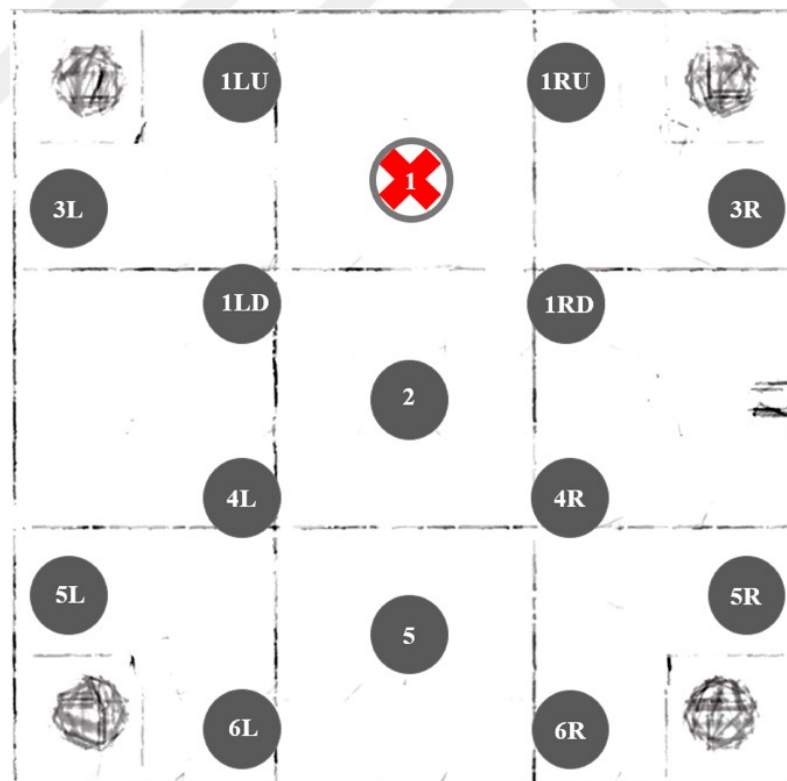


Figure 4.7 : Positions determined for roughness range determination across the build plate considering distance to laser and left-right side symmetry. Position 1 is directly under the laser and as the numbers increase, distance to laser (red x) increases and L and R letters refer to left and right sides respectively.

In the group, orientation 1 is in parallel to the laser beam whereas orientation 4 is perpendicular to the laser beam (see Figure 4.9). Orientations 2 and 3 are rotated 45 degrees in opposite directions. And finally, orientation 5 and 6 are to test angled parts (60°) that are parallel and perpendicular to the laser beam respectively. Since there are different orientations, front and back sides for parts are different but number of orientations in red in Figure 4.7 are placed at the front side of every orientation and measured values are recorded accordingly.

Surface roughness measurement results are compared only for Ra values and listed in Table 4.5. 541 measurements are done (3 measurements for each side of every sample) and averaged front and back results for different orientations and positions are compared.

Table 4.5 : Roughness measurement results across the build chamber for 15 positions and 6 orientations.

Position	Orientation	Front			Back		
		Avg Ra	Avg Rz	Avg Rt	Avg Ra	Avg Rz	Avg Rt
1	1	7,48	45,47	53,70	7,95	48,97	59,23
1LU	1	8,05	49,13	59,27	8,52	55,13	72,60
1LD	1	7,26	42,75	51,40	8,92	61,57	81,80
1RU	1	15,65	88,80	106,10	7,11	45,97	55,60
1RD	1	7,64	45,47	54,85	7,81	46,59	54,63
2	1	6,94	42,83	53,61	8,22	50,82	64,28
3L	1	7,55	45,87	51,98	10,11	63,44	86,22
3R	1	17,72	97,27	121,08	7,52	46,02	54,44
4L	1	6,74	41,31	46,51	7,67	46,93	55,69
4R	1	7,39	44,92	51,38	8,42	57,48	79,21
5L	1	7,34	43,57	51,99	8,24	52,90	68,09
5	1	7,51	45,27	54,90	8,32	51,24	59,97
5R	1	15,42	88,01	110,94	8,16	49,68	56,52
6L	1	7,12	41,67	48,87	8,58	54,97	67,77
6R	1	15,88	88,97	114,00	9,64	63,13	96,77
1	2	10,44	66,87	84,70	7,51	47,00	58,03
1LU	2	7,70	46,30	61,53	8,38	53,00	63,43
1LD	2	8,46	50,80	59,89	7,82	47,48	57,59
1RU	2	7,79	47,90	55,70	10,45	66,60	86,50
1RD	2	8,29	49,17	59,69	7,17	44,63	53,22
2	2	11,10	72,48	103,33	7,18	44,41	52,09
3L	2	6,77	43,48	52,35	8,05	48,79	58,32
3R	2	8,10	49,57	59,46	8,22	53,41	65,06
4L	2	8,21	49,60	61,10	7,10	43,65	53,34
4R	2	8,61	53,21	62,04	6,91	42,25	51,87
5L	2	8,67	57,16	75,22	7,51	45,16	53,59

Table 4.5 (continued) : Roughness measurement results across the build chamber for 15 positions and orientations.

Position	Orientation	Front			Back		
		Avg Ra	Avg Rz	Avg Rt	Avg Ra	Avg Rz	Avg Rt
5	2	11,30	68,74	92,91	7,01	43,11	51,09
5R	2	19,52	103,88	118,78	9,61	61,78	81,92
6L	2	8,15	53,44	63,79	7,27	43,90	48,91
6R	2	16,86	92,61	124,89	8,18	52,59	68,33
1	3	8,13	51,27	58,97	8,92	57,57	76,67
1LU	3	7,56	49,17	59,80	11,37	72,90	96,50
1LD	3	6,73	40,72	47,43	7,48	44,92	50,05
1RU	3	9,65	61,68	73,72	6,80	40,18	49,31
1RD	3	16,01	92,90	125,57	7,43	45,04	51,91
2	3	9,04	55,84	73,76	7,33	46,14	54,44
3L	3	7,57	46,45	54,42	8,21	50,96	60,29
3R	3	17,44	95,21	121,27	7,06	42,94	51,57
4L	3	7,28	44,39	51,56	7,66	46,84	53,69
4R	3	13,92	81,90	101,56	7,34	45,67	52,48
5L	3	7,62	45,05	54,65	8,17	45,55	50,67
5	3	14,85	81,99	98,32	7,71	47,72	61,74
5R	3	20,75	111,02	135,57	9,01	56,34	67,93
6L	3	8,36	51,65	59,34	7,91	48,37	59,49
6R	3	20,11	109,75	128,55	9,03	57,32	77,84
1	4	9,70	57,37	70,83	8,15	48,30	58,60
1LU	4	7,57	45,80	55,80	8,55	52,27	66,70
1LD	4	6,84	43,22	50,72	8,05	49,58	62,72
1RU	4	8,62	52,83	62,60	8,05	52,30	67,73
1RD	4	10,54	65,30	84,98	6,70	41,13	49,33
2	4	10,49	62,41	82,51	8,25	49,17	59,49
3L	4	7,33	44,69	52,23	8,32	56,51	70,69
3R	4	18,73	96,77	116,56	8,18	53,04	74,63
4L	4	8,23	48,26	54,39	7,91	47,05	56,92
4R	4	17,83	98,77	120,29	7,03	42,46	49,91
5L	4	8,48	52,20	63,24	8,16	51,47	67,26
5	4	16,51	93,69	117,75	7,80	49,40	62,49
5R	4	19,75	109,99	139,54	9,54	60,01	73,08
6L	4	9,50	58,15	70,87	7,99	49,84	60,08
6R	4				9,78	60,07	75,92
1	5	7,95	47,00	56,20	8,91	57,90	77,03
1LU	5	8,51	54,47	66,03	8,80	56,13	69,93
1LD	5	8,40	51,65	63,58	9,13	56,61	65,72
1RU	5	14,89	87,05	111,55	8,82	55,29	63,68
1RD	5	9,96	58,19	73,10	8,35	54,25	67,44
2	5	7,40	45,33	51,68	8,27	53,24	66,64
3L	5	8,82	53,52	66,92	9,48	57,77	67,54
3R	5	13,48	78,98	102,97	9,63	57,91	73,44

Table 4.5 (continued) : Roughness measurement results across the build chamber for 15 positions and orientations.

Position	Orientation	Front			Back		
		Avg Ra	Avg Rz	Avg Rt	Avg Ra	Avg Rz	Avg Rt
4L	5	7,92	48,41	57,33	8,64	52,92	64,35
4R	5	9,00	56,83	73,81	8,31	53,61	66,03
5L	5	8,19	47,30	53,52	8,83	57,20	74,80
5	5	8,67	54,99	68,52	9,55	57,63	68,45
5R	5	12,29	76,86	100,30	9,56	57,29	70,15
6L	5	7,72	48,38	61,08	8,83	56,42	70,90
6R	5	10,65	66,91	84,59	9,53	62,05	74,42

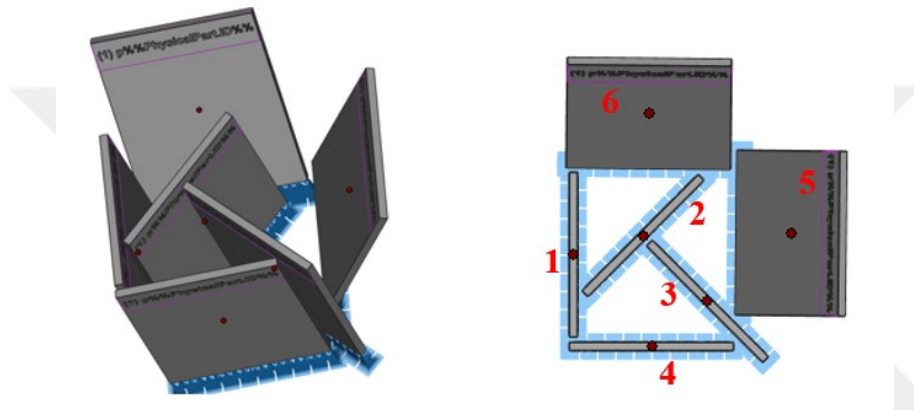


Figure 4.8 : Tilted side view of the group of 6 with six different orientation is shown on the right. Top view of group of 6 is on the left. The number of the orientation is shown with red numbers and the side that the numbers are placed are considered as front in the results.

Firstly, the common and most obvious behavior across the build chamber is that the samples in all orientations that are at the right side positions have higher roughness values than the ones on the left side positions. While the Ra values for right side are between 6-9 μm , for left side, the range increases to 7-20 μm . The main variety between left and right side of the chamber is the recoater and Ar flow moving from right to left and the scanning done from left to right. Hitzler et al stated that there is no significant effect of Ar flow for the difference between sides of one sample that are parallel or averting to the flow, but flow might have a bigger effect when left and right sides are compared across the whole chamber [41]. Another explanation might be the instant heat difference between right and left sides. Since the scanning across the build plate is completed from left to right and new powder layer is spread by the recoater from right to left, relatively hotter parts are subjected to new powder at the right side which can cause extra semi-attached particles on the surface.

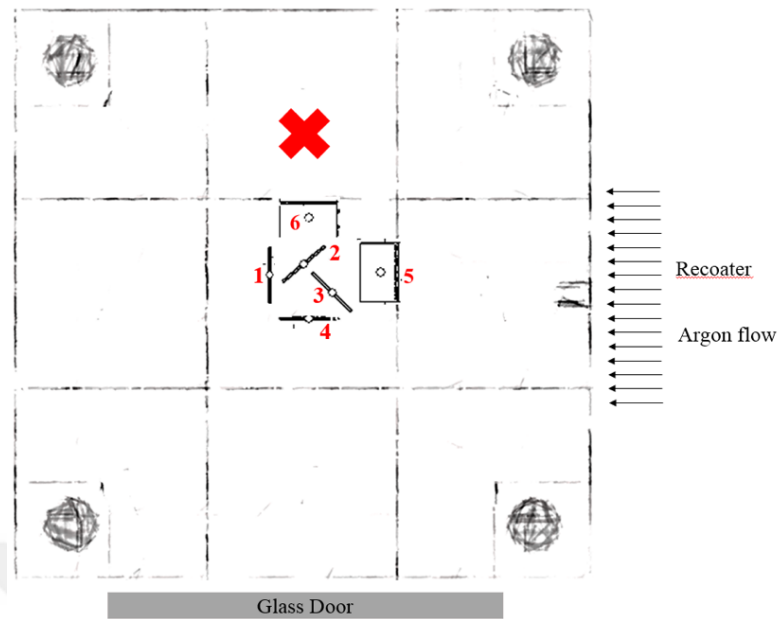


Figure 4.9 : Orientation 1 and 5 parallel to laser beam (perpendicular to the recoater and Ar flow), orientation 4 and 6 are perpendicular to laser beam and orientation 2 and 3 are 45° rotated.

Another observation generally seen across the build chamber, roughness values measured for the sides facing the door are increased as the distance from the laser increases (position 4-5-6). However, Hitzler stated that the door oriented faces have lower roughness for their samples [41]. The inert gas stream and the door are in the same position with this study. However, they stated that considering two-way recoating procedure applied, this result was not expected and the reason is not clarified in the paper. Also, the laser location and process parameters which have an effect on roughness are not stated so these should be considered for the difference between two studies. On the other hand, for this study, even with different orientations, door facing sides have higher surface roughness. This might be because of the heat distribution difference in the area close to glass door. However, this effect is mentionable only for the right side and in the middle (except for orientations in the middle that parts are perpendicular to the door) of the chamber, not for the left side. Therefore, the previous possible effects for left and right difference seem more influential on surface roughness across the chamber.

For the angled parts (orientations 5 and 6), rather than front and back, there are downward) and upward sides. For orientation 6; downward sides are facing the laser while for orientation 5; since it is parallel to the laser beam, there is no specific side

that is facing the laser (as in the case of orientation 1) (see Figure 4.8). For orientation 6, downward sides have lower surface roughness values. However, Kleszczynski et al. showed that downward sides have higher surface roughness stating that the only variable was the laser incidence angle. Laser beam is circular only in the middle of the chamber and as the position changes beam becomes more elliptical. Also for the upward and downward sides (angled structure), laser incidence angle varies as can be seen in Figure 4.10. It is suggested that difference in incidence angle may cause the difference in focus and energy input. In the figure, while c and a are shown as laser facing surfaces, b and d are facing away from the laser. Enlarged view of the difference between c and d shows that laser beam with lower angle can cause instable wetting which leads to a wider meltpool through the underlying powder bed and increased semi-attached particles on surface d may increase surface roughness. However, for higher angles, beam effects smaller region that is covered by the bulk material for surface c.

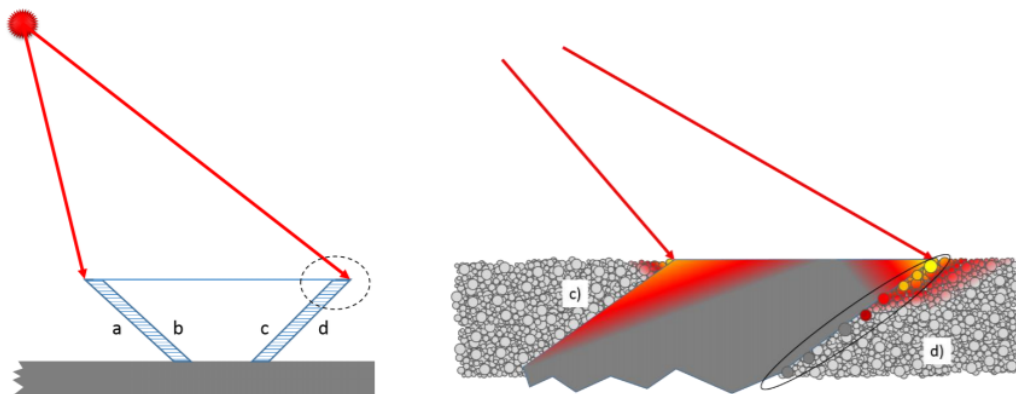


Figure 4.10 : Due to the angled structure, laser incidence angle differs for surfaces regarding to facing the laser or not. While c and a are laser facing surfaces, b and d are facing away from the laser. On the right, enlarged view of the difference between c and d shows that instable wetting causes semi-attached particles on surface d whereas it only effects bulk material for surface c [5].

In the current study, on the other hand, except for the right bottom of the build chamber there is no significant difference between upward and downward sides for angled parts. Roughness values for two sides of angled parts are quite similar especially for orientation 5 and the high difference is only observed at the right bottom of build chamber for orientation 6. However, the fact that there is no significant difference between upward and downward sides can not be explained yet. When different orientations for vertical samples are examined, in addition to the above conclusions, especially in the middle positions, it can be clearly seen that the

minimum front and back differences are displayed for orientation 1 and the maximum differences are recorded for orientation 6. While the front and back Ra values are almost same (differences smaller than 2 μm might be explained by measurement error) for orientation 1, the difference increases to 3-7 μm for orientations 2 and 3 and 11 μm for orientation 6. Even it was expected that there will be a circler effect of laser across the chamber, the same orientations belong to different groups of six (simply rotated to left and right as a group), do not show the same behavior. Therefore, for future examinations, to examine the effect of orientation on roughness for several positions, parts should be placed on different positions defined by the function of x or y rather than circler rotations as a function of radius (distance from the laser).

As conclusion, there is significant surface roughness increase for right side of the chamber which increases further when the distance from the laser increases. The significant increase in surface roughness is seen only on the sides that are facing away from the laser. Therefore, by the increased surface roughness, the difference between laser facing and the other side increases. Angled and vertical parts show a difference in surface roughness even when they both are placed parallel to the laser beam. Angled parts parallel to the beam have lower roughness compared to vertical ones. On the other hand, both for the angled and vertical parts that are placed perpendicular to the beam, high surface roughness values (also high difference between front and back) are measured.

Therefore, in order to see the effect of surface roughness on mechanical behavior, to eliminate the effect of orientation and overhang angle, vertical design in orientation 1 and 5 positions representing min max and intermediate roughness ranges (1LD, 2, 4R, 5R and 6R in Figure 4.11) are selected for the following section.

4.3 Mechanical Characterization

For mechanical characterization a 3rd and final build is prepared based on the roughness characterization input. 5 of 15 positions that are examined in the previous chapter are selected considering having samples of minimum, maximum and intermediate roughness ranges across the build chamber as shown in Figure 4.11. Positions are named from 1 to 5 according to their distances to laser and roughness

ranges where position 1 has the minimum and position 5 has the maximum roughness range according to the previous part.

Tensile bar specimens are printed as previously designed (see Material and Methods) to be able to apply mechanical tests directly and placed onto 5 positions vertically with the same orientation; front facing away from the laser and back facing the laser.

For all samples, before mechanical testing, microstructure is examined, surface roughness, width and thickness are measured.

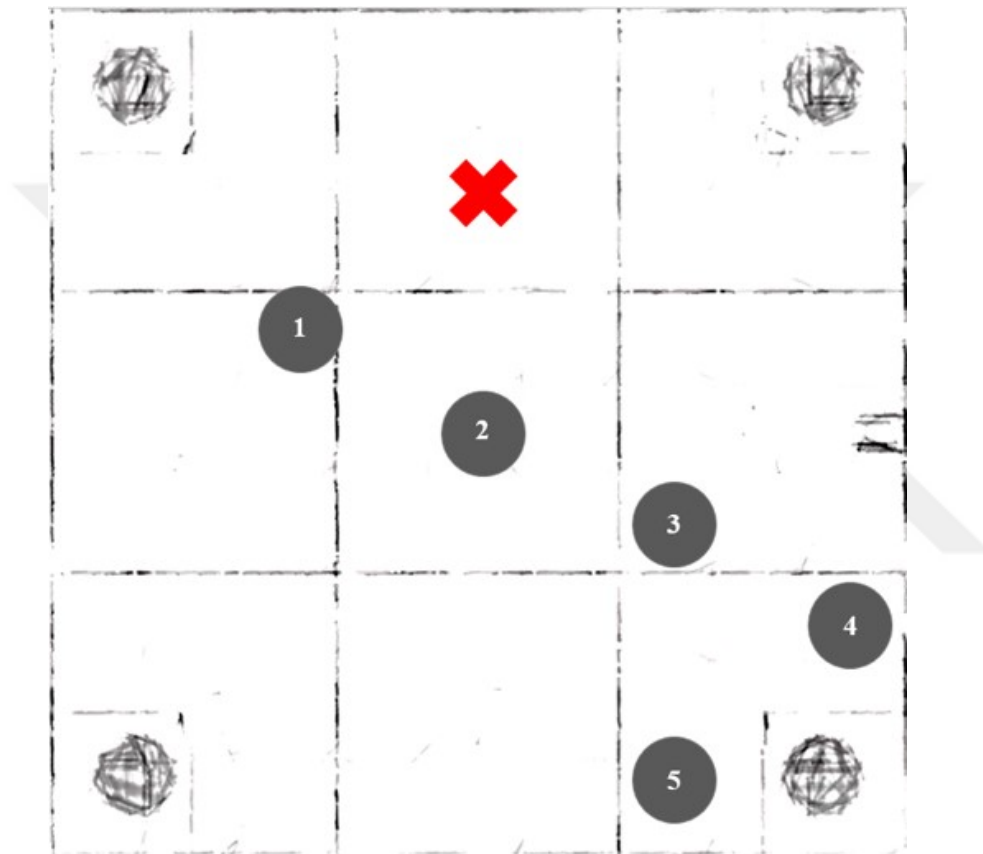


Figure 4.11 : 5 positions selected across the chamber. Laser location is shown via red x and positions are shown with grey spheres and named 1-5. Position 1 has the minimum roughness range whereas position 5 has the highest.

4.3.1 Microstructure

Since different locations across the build chamber is used, samples of each case that is used in this work are examined under optical microscopy.

Microstructures of as-built cpTi samples from positions 1 and 5 viewed along the axis perpendicular to the building direction (xy cross section) are shown in Figure 4.12 and 4.13, respectively. The structure is visible at 20x and 50x magnification.

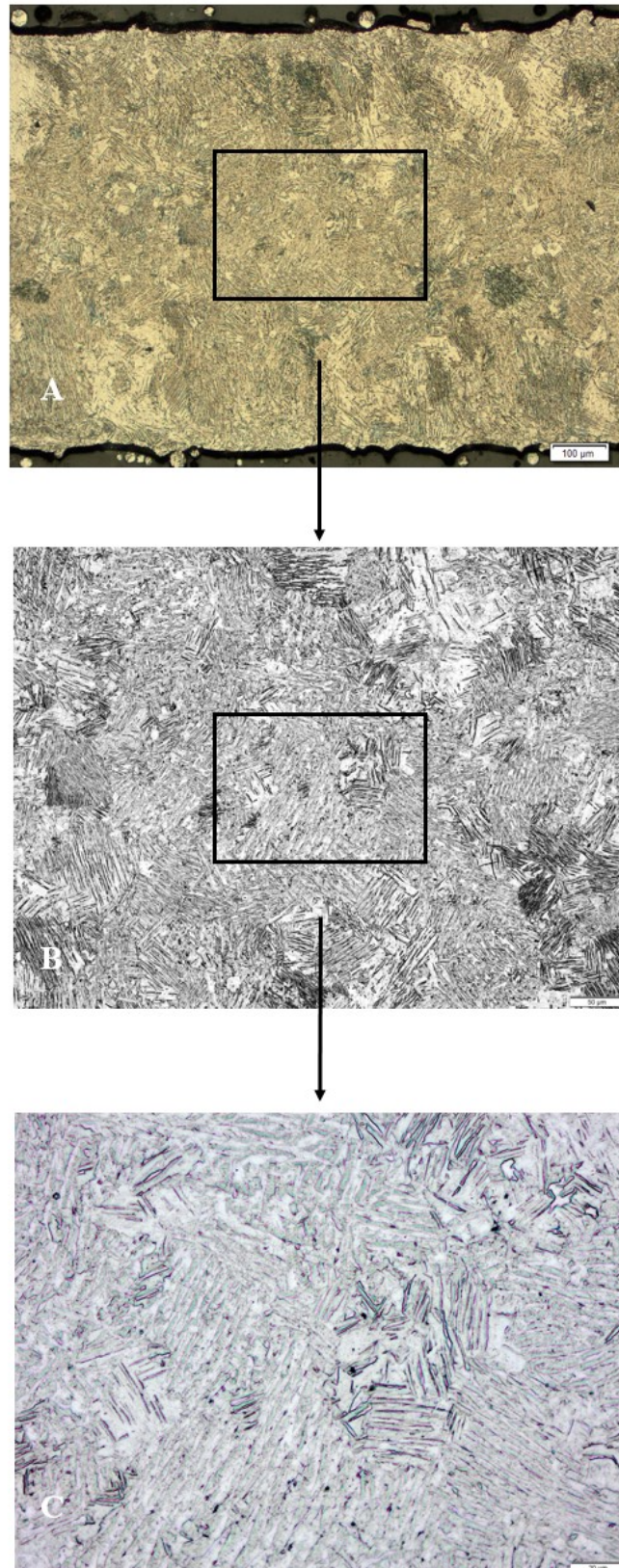


Figure 4.12 : Optical microscopy images for sample from position 1 (minimum surface roughness) A) 10x, B) 20x and C) 50x.

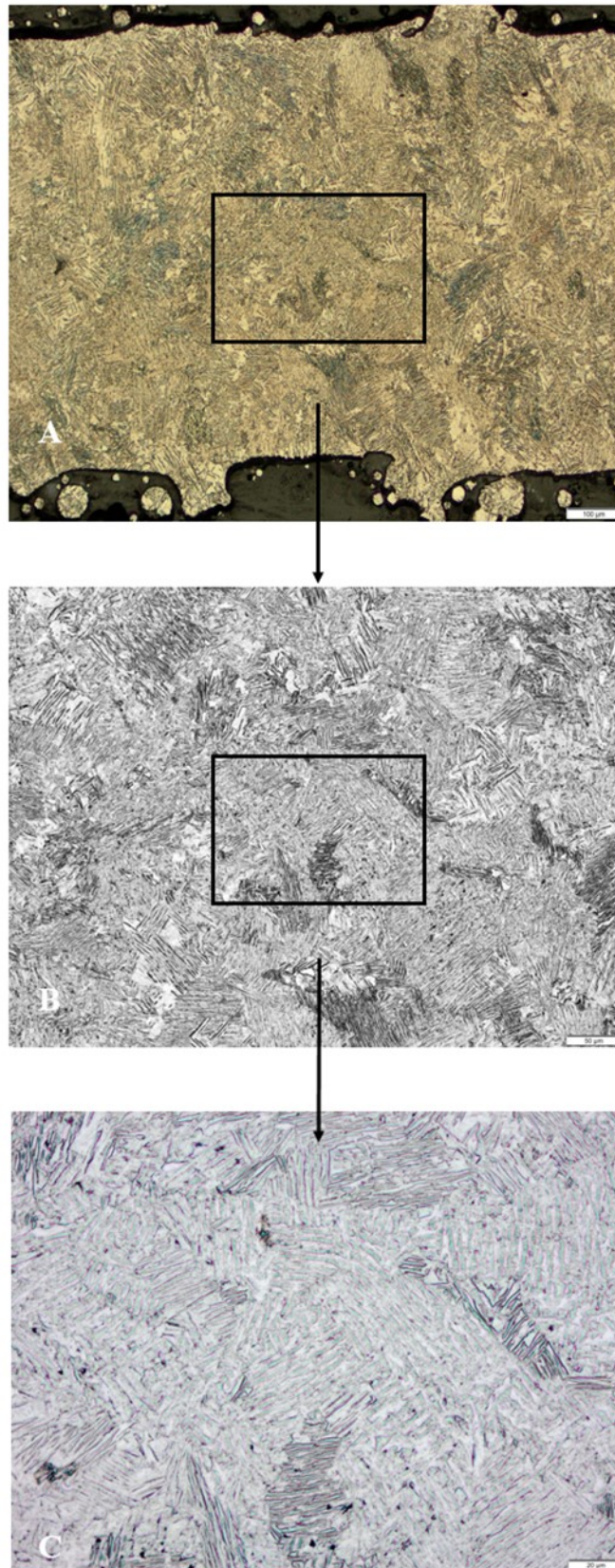


Figure 4.13 : Optical microscopy images for sample from position 5 (maximum surface roughness) A) 10x, B) 20x and C) 50x.

As can be seen from the figures, there is no mentionable difference between the microstructures obtained in different locations. Both structures show typical SLM produced cpTi properties as in prior-beta grain structure with fine acicular alpha. As a result of rapid solidification of β phase in melted metal during SLM, large amount of acicular structure occurs. Since the microstructure is same, it is not considered as a possible cause for different behaviors.

4.3.2 Roughness measurement

4.3.2.1 As-Built

In Table 4.6, surface roughness values are listed for the 5 positions. For every position, 15 samples are measured and the average Ra, Rz and Rt results of the positions are given in Table 4.6.

Table 4.6 : The average Ra (μm), Rz (μm) and Rt (μm) results with standard deviations (STDs) of 15AB samples for 5 different positions grouped for front and back sides.

Status	Position	Avg Front			Avg Back		
		Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)
AB	1	10,22 $\pm 0,89$	61,16 $\pm 5,85$	75,81 $\pm 10,35$	8,55 $\pm 0,40$	50,03 $\pm 7,58$	62,31 $\pm 6,47$
AB	2	10,92 $\pm 1,55$	65,40 $\pm 10,36$	81,57 $\pm 16,56$	8,32 $\pm 0,54$	48,40 $\pm 4,55$	57,01 $\pm 5,70$
AB	3	16,46 $\pm 1,76$	89,15 $\pm 6,66$	106,66 $\pm 6,94$	9,01 $\pm 0,49$	53,95 $\pm 4,38$	67,61 $\pm 8,88$
AB	4	16,51 $\pm 2,46$	91,19 $\pm 10,20$	112,35 $\pm 14,44$	7,56 $\pm 0,37$	45,73 $\pm 3,27$	55,26 $\pm 5,43$
AB	5	20,96 $\pm 1,69$	111,97 $\pm 7,20$	130,93 $\pm 9,46$	8,71 $\pm 0,76$	54,01 $\pm 5,19$	67,47 $\pm 8,51$

As can be seen the average back side roughness values are similar for every position while the average front values for all Ra, Rz and Rt increase almost 100%. Ra range for position 1 and 2 are similar and defined around 10 μm . Positions 3 and 4 are also showing similar results and the Ra values are around 16 μm whereas position 5 has the highest roughness as expected and Ra values are around 20 μm . Rz and Rt values are also displaying the same trend and increase from position 1 to position 5.

4.3.2.2 Post-processed

Since position 5 has the highest surface roughness, it is chosen to investigate the effect of standard Materialise Post Processing (MPP) on this position. Surface roughness, thickness (t) and width (w) are measured after MPP is applied and results are shown in Table 4.7.

Table 4.7 : The average Ra (μm), Rz (μm) and Rt (μm) with STDs, thickness (mm) and width (mm) results of 19 MPP samples for the position 5 grouped as front and back sides.

Status	Position	Avg Front			Avg Back			Avg T	Avg W
		Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)	mm	mm
Before MPP	5	21,15 $\pm 1,09$	111,95 $\pm 5,12$	131,68 $\pm 7,80$	9,01 $\pm 0,69$	54,52 $\pm 5,49$	66,14 $\pm 9,18$	0,985	4,023
After MPP	5	12,85 $\pm 1,19$	69,16 $\pm 6,57$	92,18 $\pm 12,91$	4,34 $\pm 0,56$	26,98 $\pm 3,27$	35,06 $\pm 5,88$	0,881	3,981

After MPP, the average Ra value decreases from 21 to 12 μm and 9 to 4 μm for front and back sides, respectively. Also Rz and Rt values decrease for both sides as well.

Another group of samples from position 4 is subjected to only the first stages of MPP which are SB1 + SB2 (see Material and Methods) and their roughness, thickness and width measurement results before and after SB are shown in Table 4.8.

Table 4.8 : The average Ra (μm), Rz (μm) and Rt (μm) with STDs, thickness (mm) and width (mm) results of 19 SB samples for the position 4 grouped as front and back sides.

Status	Position	Avg Front			Avg Back			Avg T	Avg W
		Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)	mm	mm
Before SB	4	16,23 $\pm 1,65$	87,85 $\pm 6,32$	106,72 $\pm 7,62$	8,82 $\pm 0,52$	54,10 $\pm 3,95$	68,83 $\pm 10,07$	0,991	4,023
After SB	4	7,60 $\pm 1,32$	43,08 $\pm 7,23$	58,48 $\pm 12,63$	4,32 $\pm 0,72$	25,86 $\pm 5,38$	36,81 $\pm 11,25$	0,861	3,999

As can be seen, SB steps have caused a decrease in Ra from 16 to 7 μm and 8 to 4 μm for front and back sides, respectively.

Finally as the roughness values decrease, decrease in thickness is also observed in both groups.

For tensile and fatigue test results, thickness related roughness parameter will be used for comparison. Ra is not preferred because it is arithmetical mean of the

absolute values of $Z(x)$ in a sampling length[44] so it neutralizes some outlying points and eliminates some peaks. In Figure 4.14, different surfaces with same R_a are shown and it is clear that they are not same even their R_a is similar. On the other hand, R_z and R_t are considering peaks and valleys which will give information of the profile considering peaks and valleys that may cause stress concentration and affect mechanical behavior. R_t is considering the maximum peak and valley along the evaluation length, R_z on the other hand is calculated by averaging the highest peak to valleys for the five sampling lengths (See Figure 4.15) which makes R_z more robust.

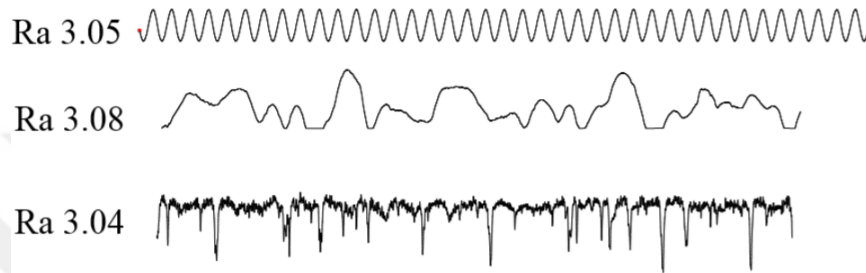


Figure 4.14 : Same R_a values are measured for different surface properties.

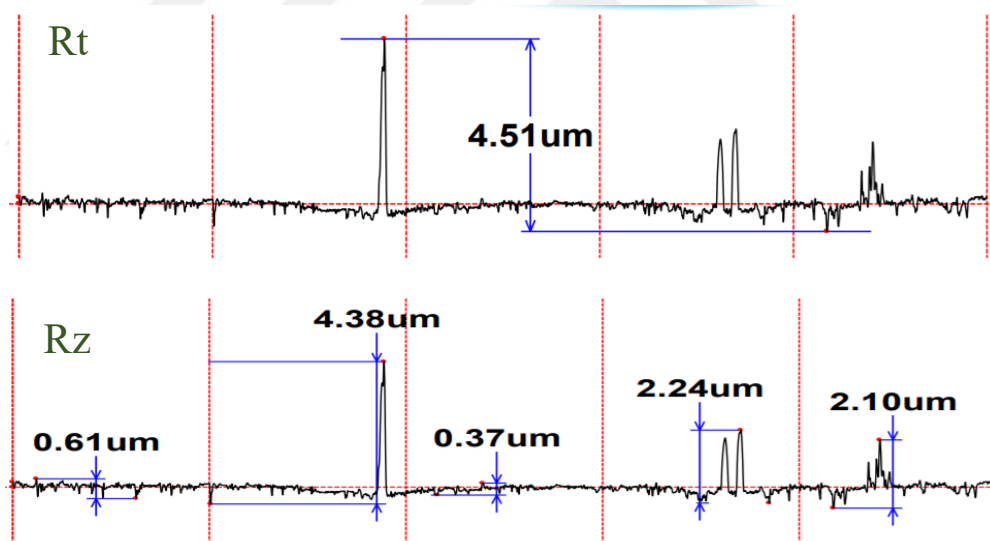


Figure 4.15 : R_t and R_z are shown on the surface profile. R_t is the highest peak to valley Average peak to valley height; averaging the highest peak to valleys for the five sampling lengths.

Therefore, while examining mechanical testing results, as roughness parameter, R_z will be used. Also, since we have different values for front and back sides of the samples, $\text{sum}R_z$ ($\text{front}R_z + \text{back}R_z$) will be used to represent every sample with one value.

4.3.3 Tensile test

4.3.3.1 As-built

For every position, 5 samples are tested. Before every test, the surface roughness, thickness (with micrometer) and width (with caliper) of the samples are measured, and the average values are listed in Table 4.9. As expected from the previous data, average front Ra values varies from 10 to 20 as position changes from 1 to 5.

The tensile tests are applied and calculations are made by cross sections calculated by multiplying measured thickness and width values shown in Table 4.9. The results of the tensile tests as in average yield strength and ultimate tensile strength are given in Table 4.10.

Table 4.9 : The average Ra (μm), Rz (μm) and Rt (μm) results with STDs, thickness (mm) and width (mm) results of 5 AB samples for 5 different positions grouped for front and back sides.

Status	Position	Front			Back			Sum	T	W
		Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)	Rz mm	mm	mm
AB	1	10,45 $\pm 0,46$	61,30 $\pm 4,73$	74,22 $\pm 7,60$	8,50 $\pm 0,50$	53,20 $\pm 2,16$	64,74 $\pm 7,04$	0,11 5	0,912	4,0 1
AB	2	12,70 $\pm 0,84$	77,10 $\pm 3,20$	97,50 $\pm 11,86$	8,53 $\pm 0,71$	51,76 $\pm 5,88$	60,60 $\pm 8,54$	0,12 9	0,916	4,0 1
AB	3	15,23 $\pm 2,15$	86,90 $\pm 8,54$	104,14 $\pm 5,87$	8,84 $\pm 0,41$	50,34 $\pm 1,65$	59,36 $\pm 4,45$	0,13 7	0,944	4,0 3
AB	4	19,15 $\pm 1,64$	102,58 $\pm 6,35$	125,80 $\pm 10,76$	7,43 $\pm 0,53$	44,50 $\pm 2,12$	56,54 $\pm 6,29$	0,14 7	0,961	4,0 3
AB	5	20,47 $\pm 2,64$	107,12 $\pm 8,80$	124,20 $\pm 8,11$	8,97 $\pm 1,25$	55,80 $\pm 4,79$	68,58 $\pm 7,52$	0,16 3	0,983	4,0 2

Table 4.10 : The avg yield strength and UTS of 5 AB samples for every 5 positions (For position 5, there was a measurement error for one sample so the results of 4 samples are averaged).

Status	Position	Avg Yield MPa	Avg UTS MPa
AB	1	493,22 $\pm 5,16$	579,35 $\pm 3,97$
AB	2	488,74 $\pm 7,02$	575,53 $\pm 5,94$
AB	3	466,26 $\pm 3,55$	548,53 $\pm 3,79$
AB	4	460,46 $\pm 6,65$	542,78 $\pm 5,03$
AB	5	448,40 $\pm 2,56$	526,95 $\pm 2,86$

It has been shown that as from position 1 to 5, surface roughness increases and results in Table 4.9 and Table 4.10 also showed that as the surface roughness increases, thickness also increases while yield strength and UTS decrease. Since

surface roughness values for front and back sides are different, sum of front and back Rz value is used for all samples to specify their roughness and represent their roughness with one value. While position 1 has an average yield strength of 493 MPa, position 5 has an average of 448 MPa so there is a 45 MPa difference between minimum and maximum roughness samples (see Figure 4.16 and 4.17). In order to determine if this is a significant effect, percentage difference calculations are done and results show that percentage difference between min and max values for measured tensile strength is 9.5% which is considered significant.

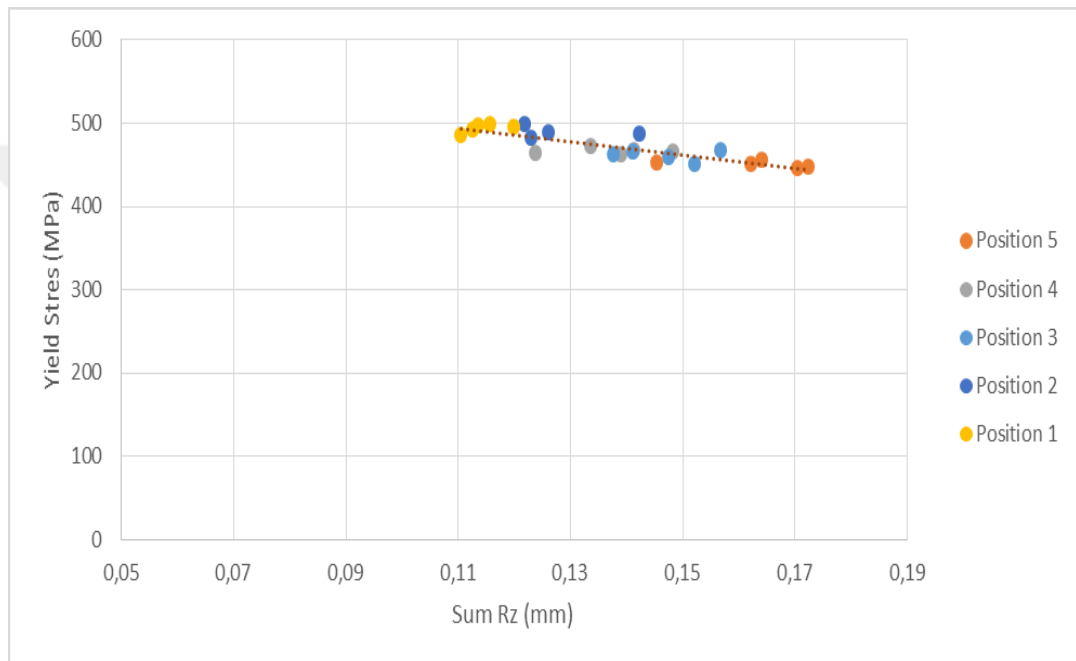


Figure 4.16 : Tensile test Yield strength (MPa) results for 25 AB for 5 different positions. Minimum roughness refers to position 1 and maximum roughness refers to position 5. Since front and back have different roughness values, sum Rz is used which refers to the sum of front side Rz and back side Rz.

It can also be seen that as the values of surface roughness parameters increase, thickness is also increasing (Table 4.9). While position 1 has an average thickness of 0,91 mm, position 5 has an average thickness of 0,98 mm. Increased thickness would lead to an increased cross section (since width of samples are almost same) which may be the reason of the difference seen between the strength calculations of high surface roughness and low surface roughness samples.

In order to have more depth information on this, relation between roughness and thickness should also be analyzed since thickness measurements are used for cross section calculations which has a direct effect on tensile test results.

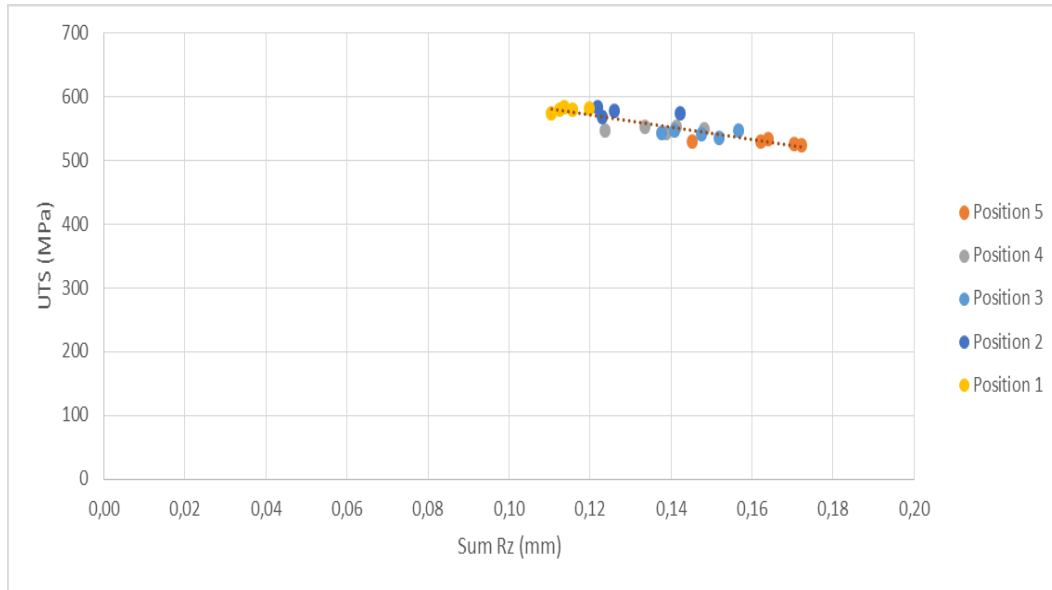


Figure 4.17 : Tensile test UTS (MPa) results for 25 AB for 5 different positions. Minimum roughness refers to position 1 and maximum roughness refers to position 5. Since front and back have different roughness values, sum Rz is used which refers to the sum of front side Rz and back side Rz.

4.3.3.2 Post-processed

Samples from position 5 that has the highest Ra values of 20 μm for front are subjected to MPP. It can be seen in Table 4.11, surface roughness values together with the thickness and width are decreased after MPP. And these MPP samples has the average Ra of 3.9 μm for back sides and the average Ra of 12.7 μm for front.

Table 4.11 : The average Ra (μm), Rz (μm) and Rt (μm) results with STDs, SumRz (mm), avg thickness (mm) and width (mm) results of 5 MPP samples of position 5 grouped for front and back sides.

		Front			Back			Sum Rz	T	W
Status		Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)	mm	mm	mm
MPP		12,71	73,06	100,44	3,94	24,60	34,32	0,098	0,88	3,97
		$\pm 0,72$	$\pm 7,48$	$\pm 10,5$	$\pm 0,13$	$\pm 1,15$	$\pm 3,48$			
AB		20,47	107,12	124,20	8,97	55,80	68,58	0,163	0,98	4,02
		$\pm 2,64$	$\pm 8,80$	$\pm 8,11$	$\pm 1,25$	$\pm 4,79$	$\pm 7,52$			

Tensile test results of MPP samples are listed as average yield strength (MPa) and average UTS (MPa) in Table 4.12 and AB sample tensile results for the same position is also listed for comparison. The average yield and UTS are higher for MPP samples which is expected due to lower surface roughness values. Also the average yield strength value for MPP samples (500 MPa) is close to the average yield for position 1 value (493 MPa) as it is for surface roughness values (see also Table 4.10).

Table 4.12 : The avg yield strength and UTS (MPa) of MPP and AB samples for position 5.

Status	Position	Avg Yield	Avg UTS
MPP	5	500,30±3,31	598,10±6,33
AB	5	448,40±2,56	526,95±2,86

Samples from position 4 that have the Ra values of 15 µm for front are subjected to only first stages of MPP which are SB1 +SB2. It can be seen in Table 4.13, surface roughness values together with the thickness and width are decreased after SB as MPP. However, SB samples has the average Ra of 3.6 µm for back sides and the average Ra of 6.2 µm for front.

Table 4.13 : The average Ra (µm), Rz (µm) and Rt (µm) results with STDs, SumRz (mm), avg thickness (mm) and width (mm) results of 5 SB samples of position 4 grouped for front and back sides.

Front				Back			SumRz	T	W
Status	Ra (µm)	Rz (µm)	Rt (µm)	Ra (µm)	Rz (µm)	Rt (µm)	mm	mm	mm
SB	6,26	36,34	47,78	3,62	21,66	30,00	0,058	0,851	4,00
	±0,75	±3,38	±7,02	±0,62	±2,90	±6,31			
AB	19,15	102,58	125,80	7,43	44,50	56,54	0,147	0,961	4,03
	±1,64	±6,35	±10,76	±0,53	±2,12	±6,29			

Tensile test results of SB samples are listed as average yield strength (MPa) and average UTS (MPa) in Table 4.14 and AB sample tensile results for the same position is also listed for comparison. The average yield and UTS are high for SB samples which is also expected due to lower surface roughness values. Also, the average yield strength value for MPP samples (500 MPa) is lower than the average yield for SB samples (514 MPa) which might also be due to relatively lower roughness of SB samples.

Table 4.14 : The avg yield strength and UTS of MPP and AB samples for position 5.

Status	Position	Avg Yield	Avg UTS
SB	4	514,21±10,50	629,71±1,25
AB	4	460,46±6,65	542,78±5,03

The percentage differences between MPP and AB is calculated as 10.9% and it is calculated as 11% for SB and AB samples

Yield strength and UTS results for MPP and SB samples are shown in Figure 4.18 and 4.19, respectively.

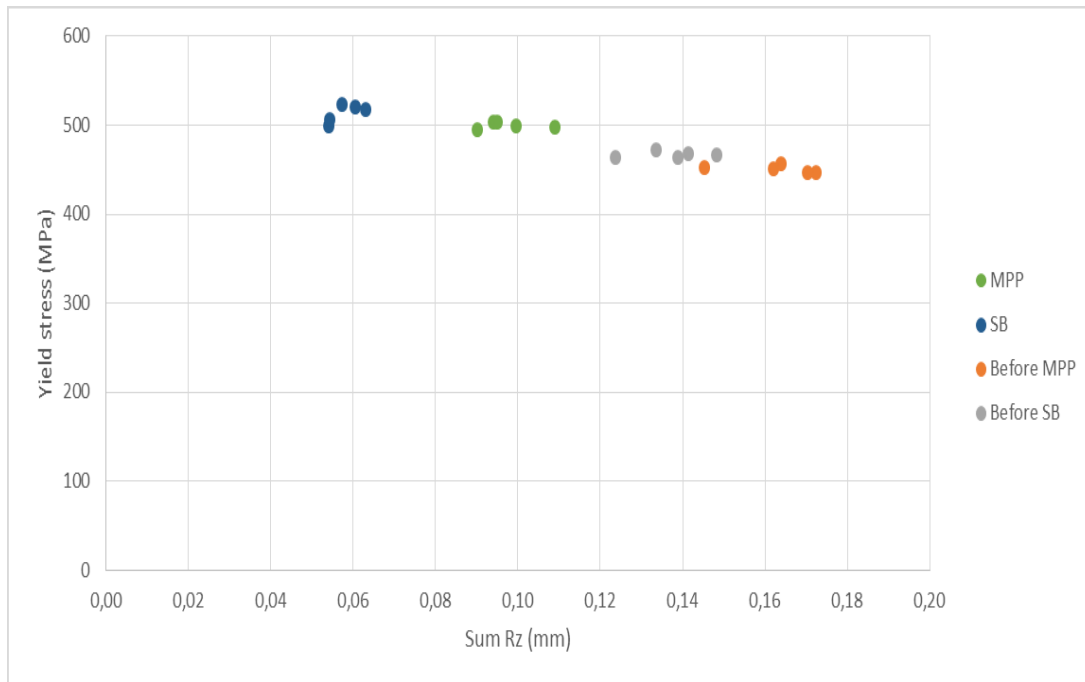


Figure 4.18 : Tensile test Yield strength (MPa) results for MPP samples which are originally from Position 5 (Before MPP) and SB samples which are originally from Position 4 (Before SB). Since front and back have different roughness values, sum Rz is used which refers to the sum of front side Rz and back side Rz.

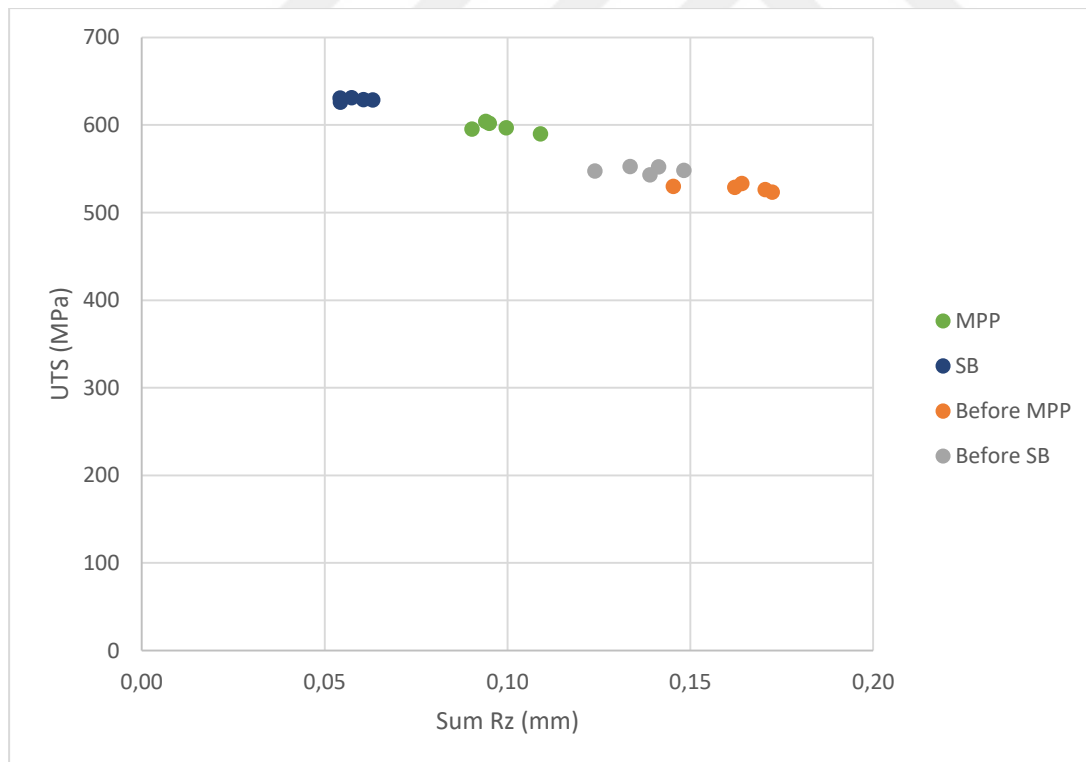


Figure 4.19 : Tensile test UTS (MPa) results for MPP samples which are originally from Position 5 and SB samples which are originally from Position 4. Since front and back have different roughness values, sum Rz is used which refers to the sum of front side Rz and back side Rz.

4.3.4 Fatigue test

For determining the fatigue test stress levels, tensile test results are used. The average yield strength of the maximum roughness group is determined as 450 MPa and it is used to calculate maximum stress level to start the fatigue program. Therefore, 80% of 450 MPa (360 MPa) is tried as the first maximum stress level. Then different percentages of this are used and stress levels are determined as 450, 360, 292, 225 and 180 MPa respectively. Considering the fact that the average yield strengths of the samples are close to each other for all groups and to be able to compare different surface roughness values, these same stress levels are used for all samples.

4.3.4.1 As-Built

For the AB samples, the yield strength showed 55 MPa difference between min and max surface roughness. To be able to see the extreme situation, samples from min and max surface roughness positions (position 1 and 5) are subjected to fatigue testing.

Roughness, thickness and width of samples are measured and average yield stress for each group is considered as the yield stress as shown in Table 4.15.

Table 4.15 : The average Ra (μm), Rz (μm) and Rt (μm) results with STDs, SumRz (mm), avg thickness (mm), width (mm) and yield stress results of AB samples of position 1 and 5 grouped for front and back sides.

Positi on	Avg Front			Avg Back			Sum Rz	Avg T	Avg W	Avg Yield
	Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)	mm	mm	mm	MPa
1	10,21	61,91	77,85	8,72	49,49	61,71	0,11	0,91	4,03	493,22
	$\pm 1,08$	$\pm 6,99$	$\pm 10,7$	$\pm 0,49$	$\pm 8,03$	$\pm 5,58$				
5	21,12	114,3	135,1	8,69	54,23	68,67	0,17	0,98	4,03	449,88
	$\pm 1,07$	$\pm 4,64$	$\pm 9,05$	$\pm 0,48$	$\pm 5,86$	$\pm 9,38$				

Determined stress levels are applied to all samples (see Material and Methods) and results are shown in Figure 4.20. Determined stress values are multiplied by the cross section and resultant value is used as the load input for fatigue test.

When the minimum and maximum surface roughness samples are compared, slight shift to the right is observed for decreased roughness. At the same stress levels, samples with lower surface roughness seem to have longer fatigue life.

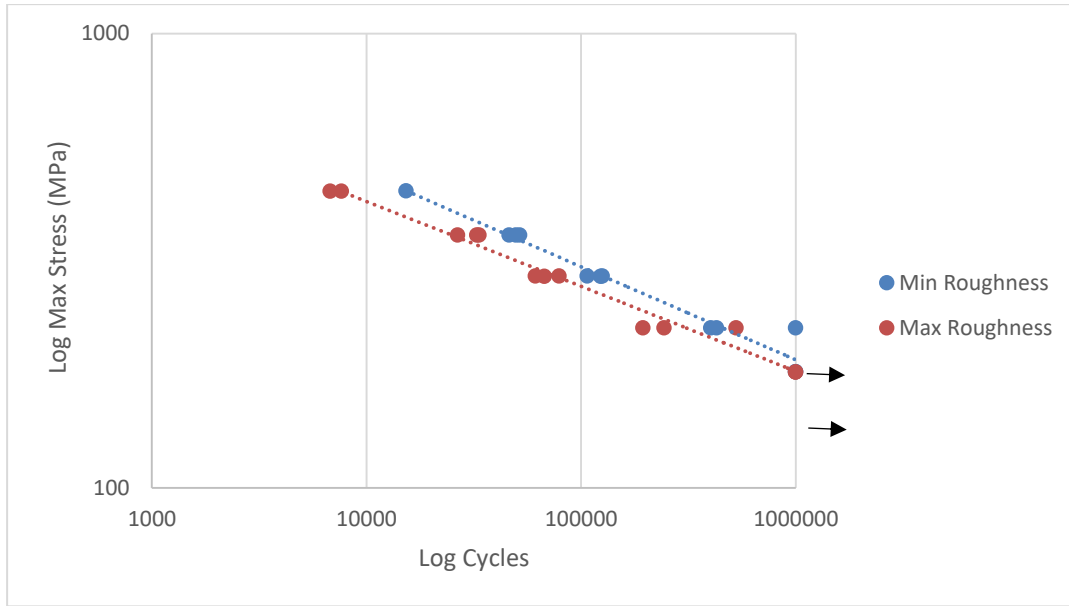


Figure 4.20 : Fatigue test results (Log-Log) for 26 AB samples tested at 5 stress levels for two different positions. Minimum roughness refers to position 1 and maximum roughness refers to position 5.

4.3.4.2 Post-processed

For the MPP samples, the yield strength is 500 MPa which means 50 MPa difference compared to AB samples on their position. Also for SB samples, yield strength increased to 514 MPa which means 15 MPa increase compared to AB samples on their position.

The results of average measured roughness, thickness, width, and yield stress for AB and MPP samples from position 5 are shown in Table 4.16.

Table 4.16 : The average Ra (μm), Rz (μm) and Rt (μm) results with STDs, SumRz (mm), avg thickness (mm), width (mm) and yield stress results of AB and MPP samples of position 5 for front and back sides.

Avg Front							Sum	Avg	Avg	Avg
Avg Back							Rz	T	W	Yield
Statu	Ra	Rz	Rt	Ra	Rz	Rt				
s	(µm)	(µm)	(µm)	(µm)	(µm)	(µm)	mm	mm	mm	MPa
MPP	13,02	68,15	90,43	4,52	28,10	35,75	0,10	0,88	3,98	499,19
	±1,31	±5,94	±12,35	±0,59	±3,35	±6,68				
AB	21,12	114,3	135,1	8,69	54,23	68,67	0,17	0,98	4,03	449,88
	±1,07	±4,64	±9,05	±0,48	±5,86	±9,38				

The results of average measured roughness, thickness, width, and yield stress for AB and SB samples from position 4 are shown in Table 4.17.

Table 4.17 : The average Ra (μm), Rz (μm) and Rt (μm) results with STDs, avg thickness (mm), width (mm) and yield stress results of AB and SB samples of position 4 for front and back sides.

Sta- tus	Posi- tion	Avg Front			Avg Back			Sum Rz	Avg T	Avg W	Avg Yield
		Ra (μm)	Rz (μm)	Rt (μm)	Ra (μm)	Rz (μm)	Rt (μm)	mm	mm	mm	MPa
SB	4	8,19 $\pm 1,12$	46,00 $\pm 6,78$	63,33 $\pm 13,11$	4,61 $\pm 0,55$	27,62 $\pm 5,22$	39,79 $\pm 11,6$	0,09	0,87	4,00	512,4
AB	4	19,15 $\pm 1,64$	102,8 $\pm 6,35$	125,80 $\pm 10,76$	7,43 $\pm 0,53$	44,50 $\pm 2,12$	56,54 $\pm 6,29$	0,11	0,96	4,03	460,4

As stated before, same stress levels are applied also to MPP and SB samples for fatigue tests and the results for AB, MPP and SB samples are shown in Figure 4.21. As can be seen easily, Figure 4.21 suggests that MPP and SB samples have longer fatigue life than AB samples for the same stress level. Also, MPP samples has longer fatigue life than SB samples as well.

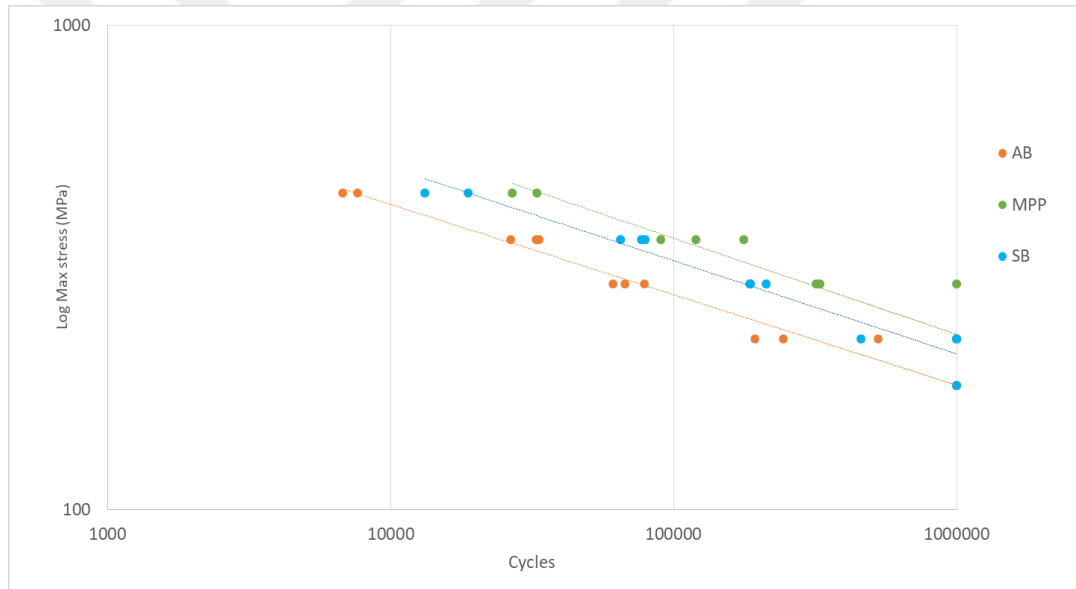


Figure 4.21 : Fatigue test results (log Max stress v. log Cycles) for 39 samples tested at 5 stress levels for 3 different groups.

4.4 Roughness and Thickness Relation

Different thickness values are measured whereas all the parts are designed to have the same thickness, 0.8 mm. It is observed that surface roughness increase is followed by thickness increase.

Generally, the thickness related roughness parameter is defined as Rt (total height) which represents “the sum of the maximum peak height and the maximum valley depth of a profile within the evaluation length” [44]. Also, Rz (max height) which

represents “the sum of the maximum peak height and maximum valley depth of a profile within the sampling length”, is a thickness related parameter [44]. In order to be able to compare thickness and R values, R values for front and back sides are added and the total R value is calculated for every sample. Therefore, the Rt that will be used means sum of Rt values for the front and back sides (Rz means sum of Rz values of front and back). Graphs shown in Figure 4.22 and 4.23 display that both ‘sum Rz’ and ‘sum Rt’ are in direct proportion to thickness.

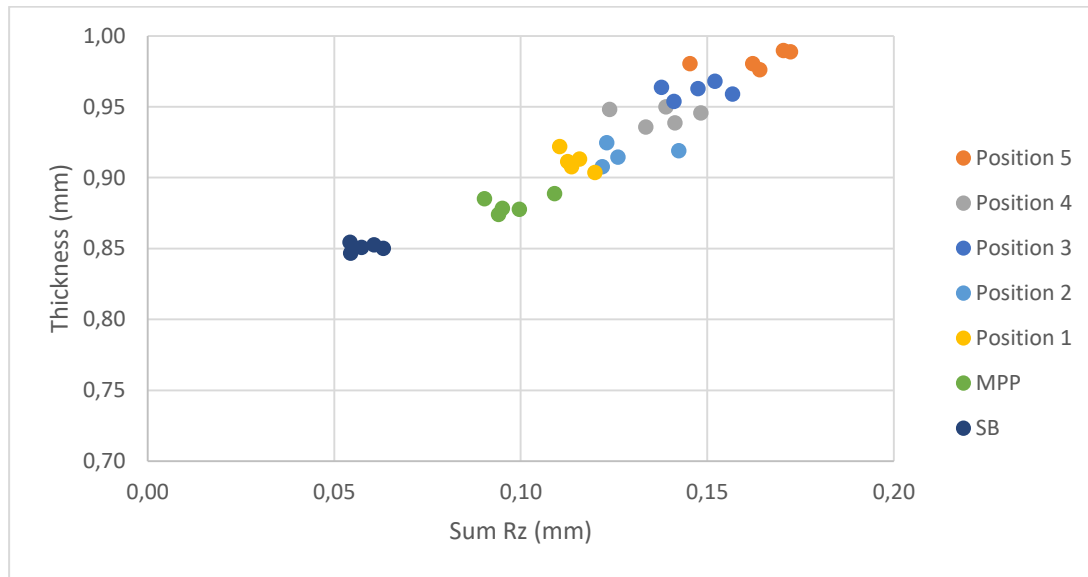


Figure 4.22 : Thickness ($t_{\text{micrometer}}$) (mm) versus Sum Rz (mm) for all 5 positions and AB, MPP and SB samples are shown. As the roughness increases (sum Rz) the thickness is also increasing.

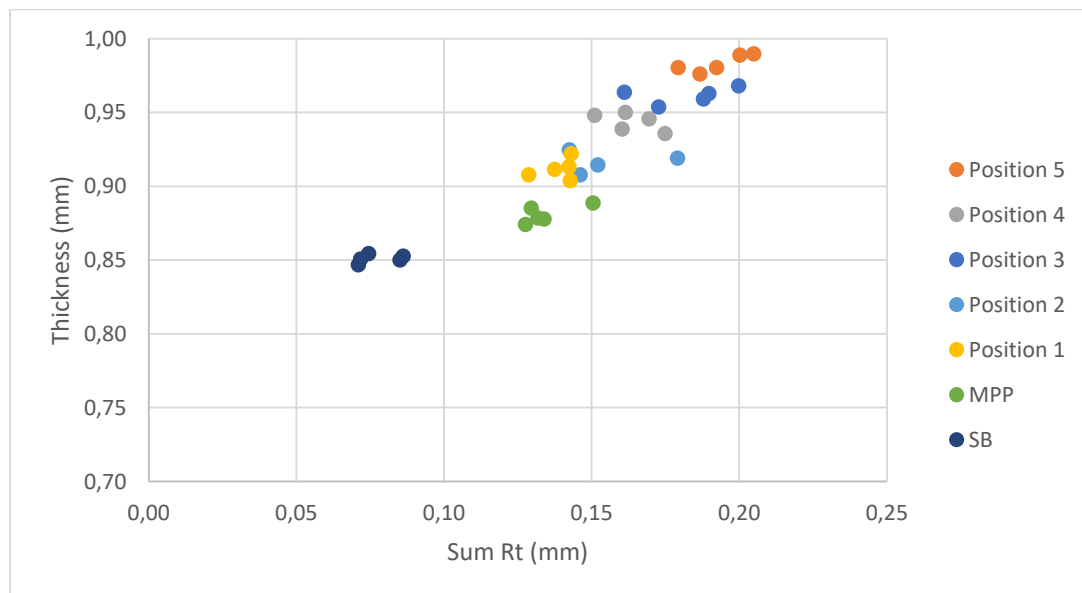


Figure 4.23 : Thickness ($t_{\text{micrometer}}$) (mm) versus Sum Rt (mm) for all 5 positions and AB, MPP and SB samples are shown. As the roughness increases (sum Rt) the thickness is also increasing.

Therefore, samples from different groups are examined in detail with optical microscopy (OM) in terms of their thickness to have further information about the roughness thickness relation.

3 samples are chosen: AB with high surface roughness, AB with low surface roughness and finally MPP. OM images for different samples are shown in Figure 4.24, 4.25, 4.26 respectively.

Firstly, in all samples it can be seen that one side is rougher than the other one which are named as front and back previously. Surface is not smooth and there are peaks and valleys along the sample which creates different roughness and thickness values.

Figure 4.24 belongs to the AB sample with high surface roughness. The horizontal white line shows the thickness that is measured considering the peaks which are caused by powder particles. The horizontal red line shows the thickness that is measured considering the valleys which can be considered as the bulk, continuous material. When the 3 measurements for each are averaged, thickness with peaks is 895 μm and thickness of bulk is 745 μm .

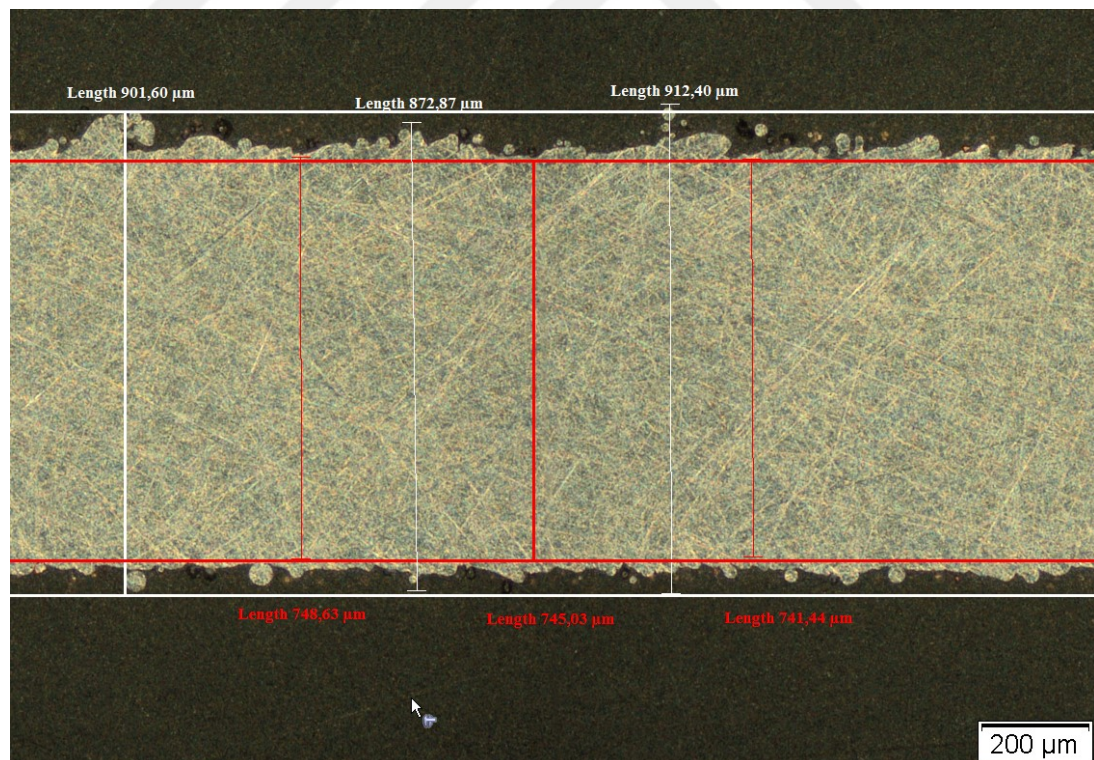


Figure 4.24 : Thickness measurement with OM for the AB high roughness sample. The horizontal white line shows the thickness measured considering the peaks which are caused by powder particles and the horizontal red line shows the thickness measured considering the valleys which can be considered as the bulk, which are averaged as 895 and 745 μm , respectively.

Figure 4.25 stands for the AB sample with low surface roughness. The red and white lines again represent the thickness considering peaks and thickness considering valleys, respectively and the average thickness with peaks equals to 862 μm whereas the thickness of bulk equals to 743 μm .

Finally PP sample is shown in Figure 4.26. Average thickness considering peaks is determined as 836 μm and average thickness of bulk is 740 μm .

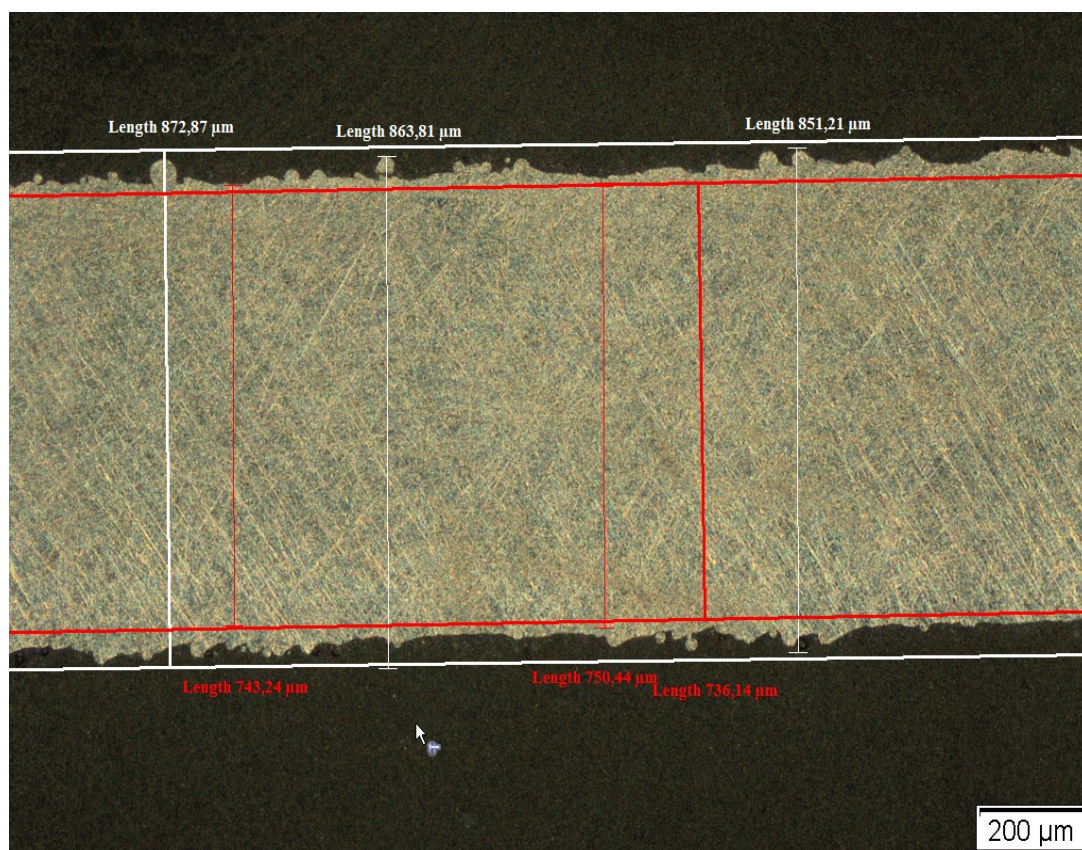


Figure 4.25 : Thickness measurement with OM for the AB low roughness sample. The horizontal white line shows the thickness measured considering the peaks which are caused by powder particles and the horizontal red line shows the thickness measured considering the valleys which can be considered as the bulk, which are averaged as 862 and 743 μm respectively.

The average thickness for bulk part equals to 0.74 mm for all the types however when the peaks are considered the average thickness values changes as 0.89, 0.86 and 0.83 mm.

Since the micrometer that is used for thickness measurement has a diameter of 6 mm, it is capable of measuring the thickness considering peaks rather than the thickness of the bulk. Therefore, as the roughness changes, peaks are changes and the thickness variation between the samples is observed.

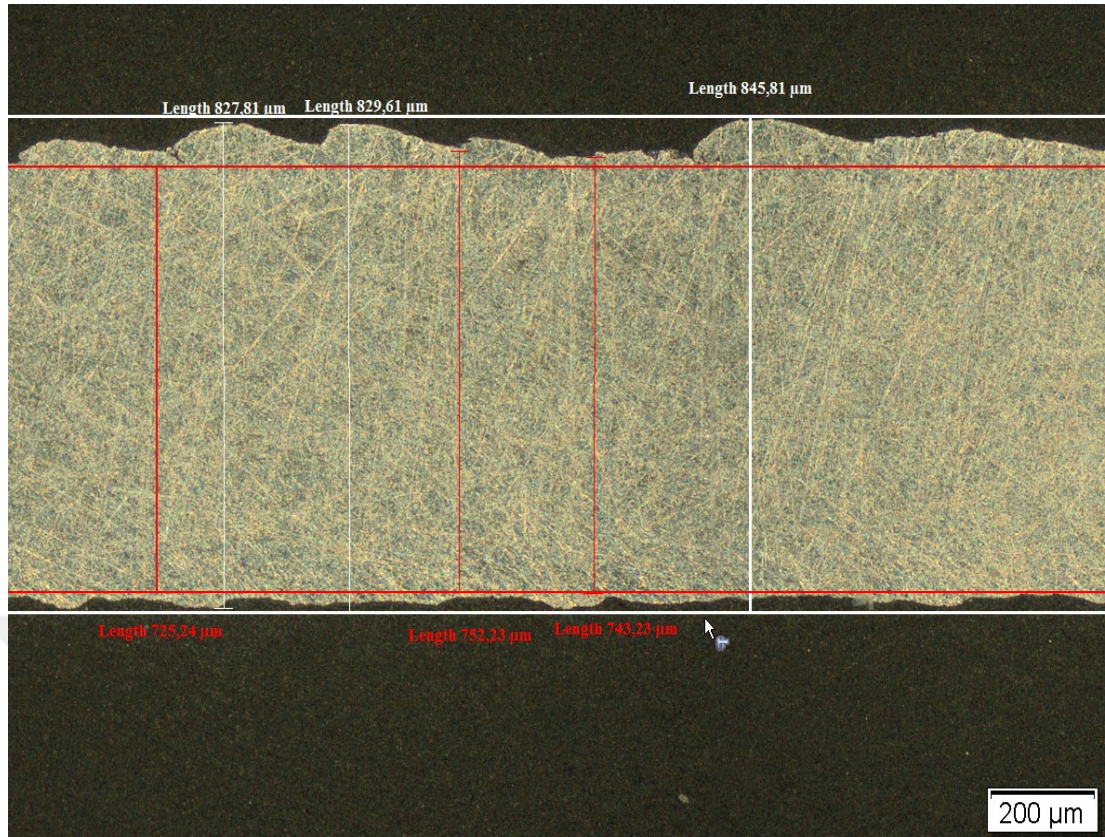


Figure 4.26 : Thickness measurement with OM for the PP sample. The horizontal white line shows the thickness measured considering the peaks which are caused by powder particles and the horizontal red line shows the thickness measured considering the valleys which can be considered as the bulk, which are averaged as 836 and 740 μm , respectively.

This assumption of thickness difference caused by the surface roughness occurred during production with attached powder particles on the surface has led to an idea of calculating theoretical “roughness free thickness”. Therefore it is assumed that our samples should have the thickness of 0.8 mm but uncontrolled roughness is added during production is also counted while measuring the thickness with micrometer ($t_{\text{micrometer}}$). In Figure 4.27, theoretical relation is also shown on the graphs as thickness that is measured equals to addition of the sum of surface roughness and the designed thickness (0.8 mm). As can be seen, ‘sum Rz’ values created less scattered data and shows a better fit to the defined theoretical relation (linear trend line) than the ‘sum Rt’ values.

When ‘t-sum Rz’ and ‘t-sum Rt’ values are examined for different positions, as can be seen in Table 4.18, the difference between measured thickness and ‘roughness free thickness’ increases as the roughness increases which is in parallel with the expectation.

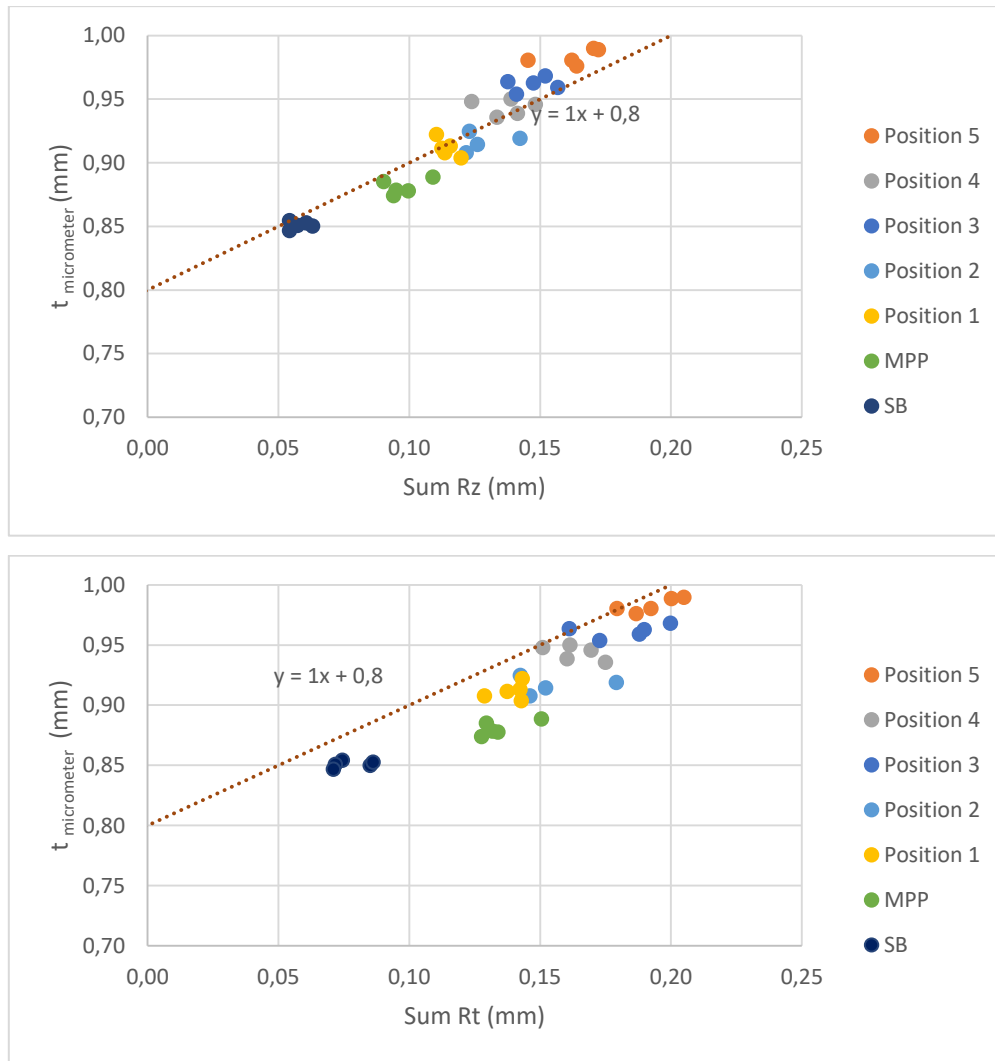


Figure 4.27 : $t_{\text{micrometer}}$ (mm) versus sum of Rz (mm) (top graph) and thickness (mm) versus sum of Rt (mm) (bottom graph) are shown. Theoretical relation as $y=1x+0,8$ is also shown considering that the designed thickness is 0.8 mm.

Table 4.18 : t-Rz and t-Rt values calculated for AB and PP tensile samples to obtain avg theoretical ‘roughness free thickness’. Delta displays the difference between measured thickness and ‘roughness free thickness’.

Status	Test	Position	t (mm)	t-Rz (mm)	t-Rt (mm)	Δ
AB	T	1	0,912	0,784	0,761	0,118- 0,151
AB	T	2	0,916	0,786	0,761	0,130- 0,155
AB	T	3	0,944	0,797	0,776	0,147- 0,168
AB	T	4	0,961	0,813	0,781	0,148- 0,180
AB	T	5	0,983	0,818	0,788	0,165- 0,195
MPP	T	5	0,881	0,783	0,746	0,098- 0,135
SB	T	4	0,851	0,793	0,773	0,058- 0,020

It can be seen from Table 4.18 that even if Rz and Rt show more significant differences in micro scale, both avg t-Rz (0,796 mm) and avg t-Rt (0,769 mm) give quite similar results (0.03 mm difference observed).

Rz has been chosen as roughness parameter (see section 4.3.2), ‘sum Rz’ is used to calculate ‘roughness free thickness’ as in ‘t-sumRz’ is defined in equation 4.1.

$$t - Rz = t_{\text{micrometer}} - (Rz_{\text{front}} + Rz_{\text{back}})$$

$$\sigma' = \frac{F}{w \cdot (t - \text{sumRz})}$$

$$\sigma = F / (w \cdot t) \quad (4.1)$$

While the OM measurements show similar bulk thickness values for all groups, there is still a difference between different groups with t-Rz calculations (roughness increases from position 1 to 5 as well as thickness and t-sumRz values). This difference can be explained due to the limitation of tactile profilometer measurements. As it is discussed previously, tactile profilometer is not capable of measuring high surface roughness values around 20 μm which requires 8 mm cut-off length. Since tactile profilometer can not reach to that point for higher roughness values, measured Rz values are lower than they should be. Therefore, the subtracted sum Rz is lower and the resultant t-sumRz thickness is higher than it should be. However, as can be seen from Figure 4.28, when the equation is applied, thickness values decrease more for rougher samples and the final thickness values are close for all samples.

Since roughness directly affects measured thickness values, while discussing the mechanical test results, ‘t-sum Rz’ as ‘roughness free thickness’ is also considered. Additionally calculated theoretical results are shown as ‘recalculated stress values’ for which roughness free thickness (t-sum Rz) is used instead of measured thickness ($t_{\text{micrometer}}$) and recalculated stress values are compared with the measured stress values. The same theory is applicable to the width as well. However, since we can not measure the surface roughness on these sides, ‘roughness free width’ is neglected for these results.

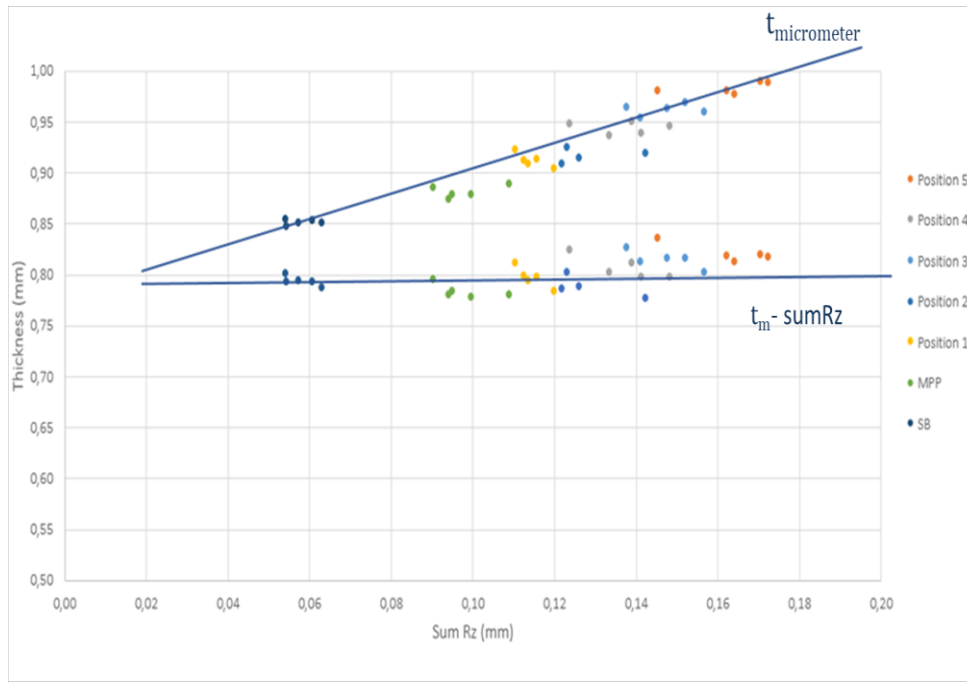


Figure 4.28 : Thickness (mm) versus sum of Rz (mm) is shown for both measured thickness ($t_{\text{micrometer}}$) values and recalculated ' $t_m - \text{sumRz}$ ' values.

4.4.1 Recalculated tensile test results

4.4.1.1 As-built samples

As listed in Table 4.19, t -Rz values are calculated for the tensile samples and inverse calculations are made; Applied loads are divided by the 'roughness free thickness' (t -Rz) values and max stress values are recalculated as shown in Figure 4.29 and Figure 4.30.

Table 4.19 : t -Rt and t -Rz values calculated for AB tensile samples of position 1 to 5 to obtain avg theoretical 'roughness free thickness'. Delta displays the difference between measured thickness and 'roughness free thickness'.

Status	Test	Position	Avg t mm	Avg t -Rz mm	Δ
AB	T	1	0,912	0,797	0,11
AB	T	2	0,916	0,788	0,12
AB	T	3	0,944	0,806	0,13
AB	T	4	0,961	0,814	0,14
AB	T	5	0,983	0,820	0,16

With the recalculation of thickness and strength, the difference between different positions that have high and low roughness values decreases. The maximum roughness samples have the average yield strength of 540 while the minimum roughness samples have the average yield strength of 568 MPa. Therefore, the

difference define as 45MPa is decreased to 28MPa. The percentage difference that is calculated for measured thickness and tensile strength values has been stated as 9.5% and significant. However, after recalculation, percentage difference decreases to a much lower value and defined as 5%.

There still is a difference but as it is discussed before, this can be explained by tactile profilometer limitation for high roughness Rz values. Also there is neglected width effect. Even if the effect of surface roughness on width couldn't be measured, when sumRz is taken as it is for thickness recalculation and 'roughness free cross section' is calculated by using 'roughness free width and thickness', the resultant curve for all samples become more straight (see Figure 4.31). Therefore neglected width effect can also cause the little difference between different positions.

For these samples, it can be said that surface roughness do not have an effect on yield strength. Thickness measurement affected by surface roughness affects the raw tensile testing results but when the roughness effect on the measurement of real cross section that is bearing the load is considered and recalculations are used for analysis, it seems that in fact surface roughness is not felt by material behavior.

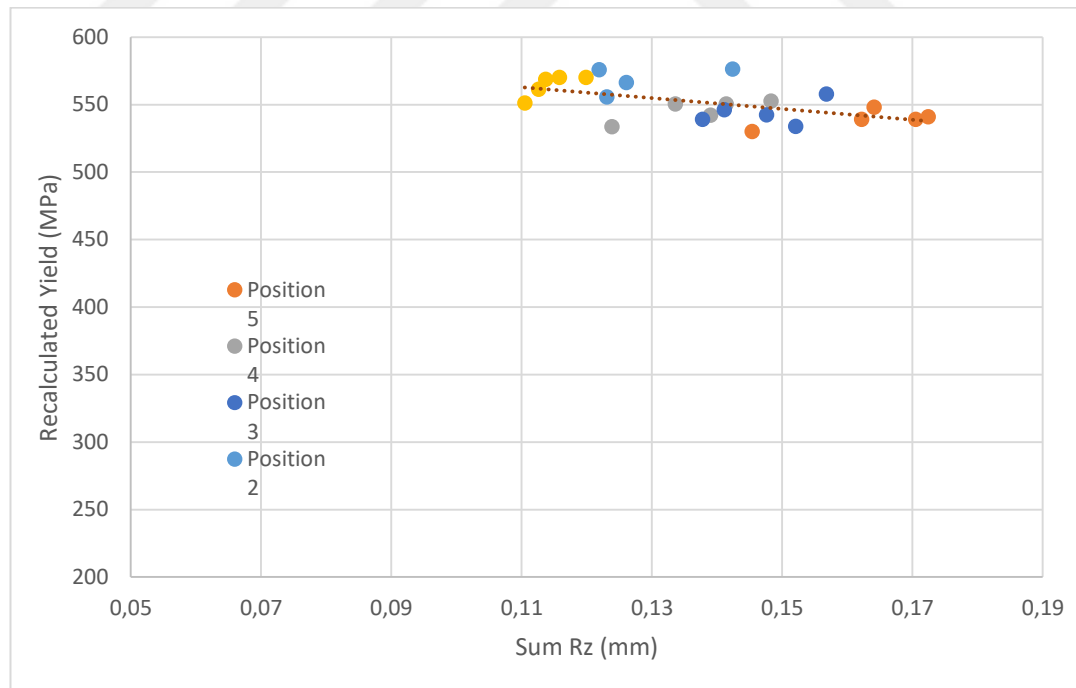


Figure 4.29 : Tensile test yield strength results with recalculated max stress for 25 AB samples for 5 different positions. Minimum roughness refers to position 1 and maximum roughness refers to position 5.

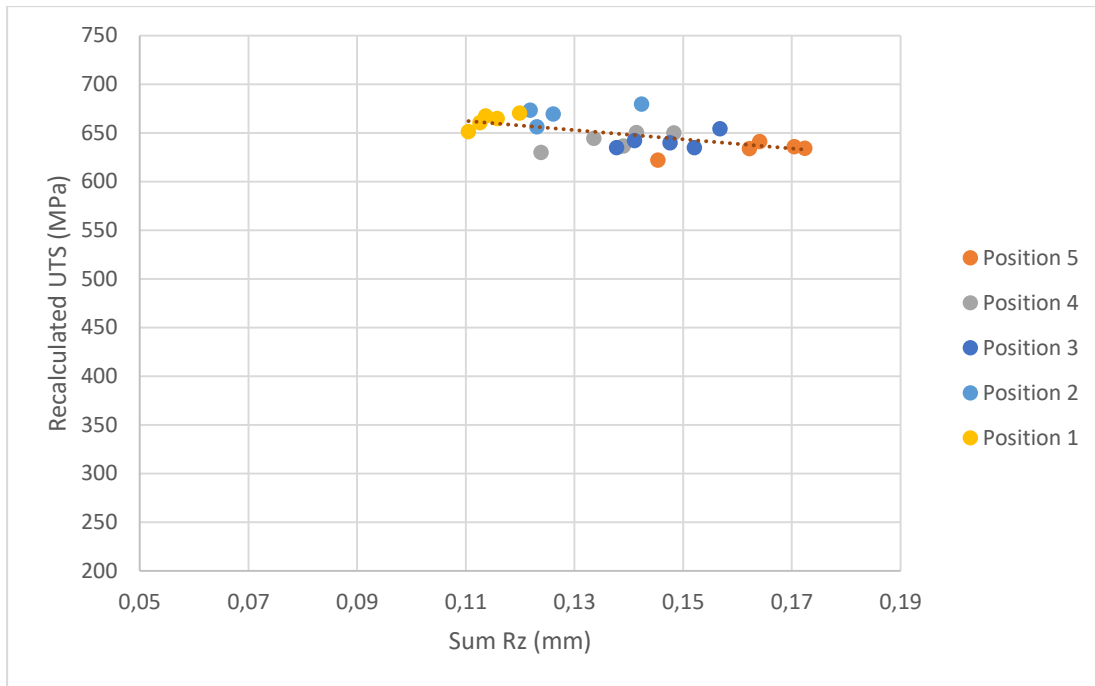


Figure 4.30 : Tensile test UTS results with recalculated max stress for 25 AB samples for 5 different positions. Minimum roughness refers to position 1 and maximum roughness refers to position 5.

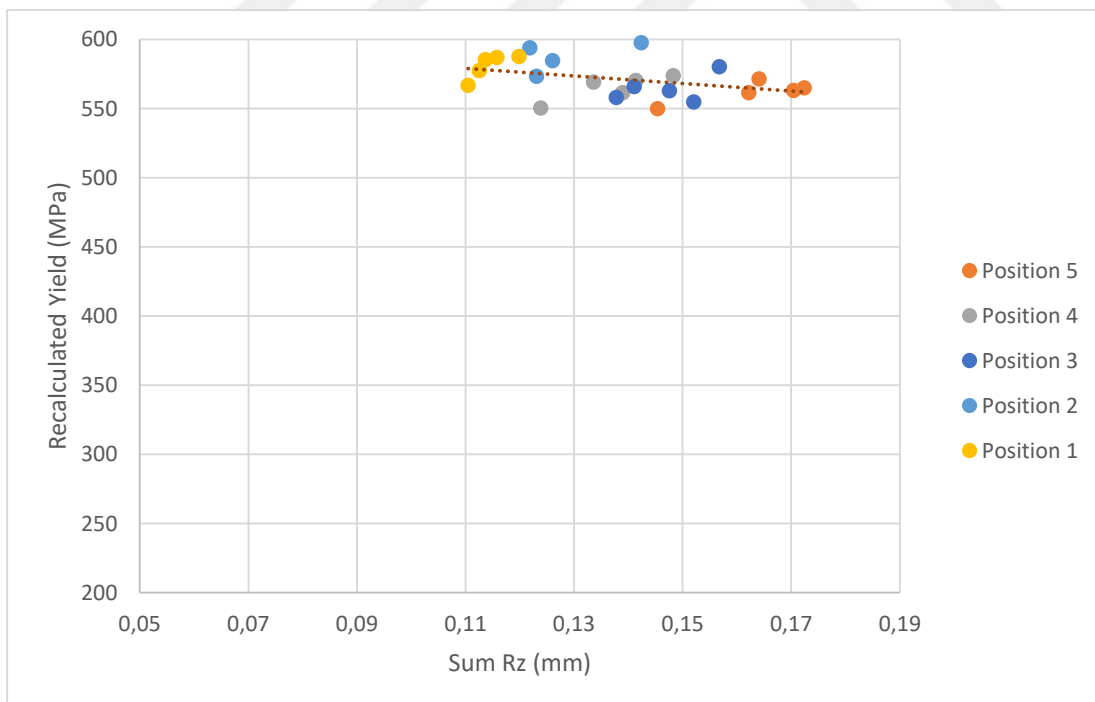


Figure 4.31 : Tensile test yield strength results with recalculated max stress for 25 AB samples for 5 different positions by using ‘roughness free thickness and width’. Minimum roughness refers to position 1 and maximum roughness refers to position 5.

4.4.1.2 Post-processed samples

As listed in Table 4.20, t-Rz values are calculated for the tensile samples and inverse calculations are made; Applied loads are divided by the ‘roughness free thickness’ (t-Rz) values and max stress values are recalculated as shown in Figure 4.32.

Table 4.20 : t-Rt and t-Rz values calculated for MPP, SB and AB tensile samples to obtain avg theoretical ‘roughness free thickness’. Delta displays the difference between measured thickness and ‘roughness free thickness’.

Status	Test	Position	Avg t mm	Avg t-Rz mm	Δ
MPP	T	5	0,881	0,780	0,10
SB	T	4	0,851	0,792	0,05
AB	T	5	0,983	0,818	0,16

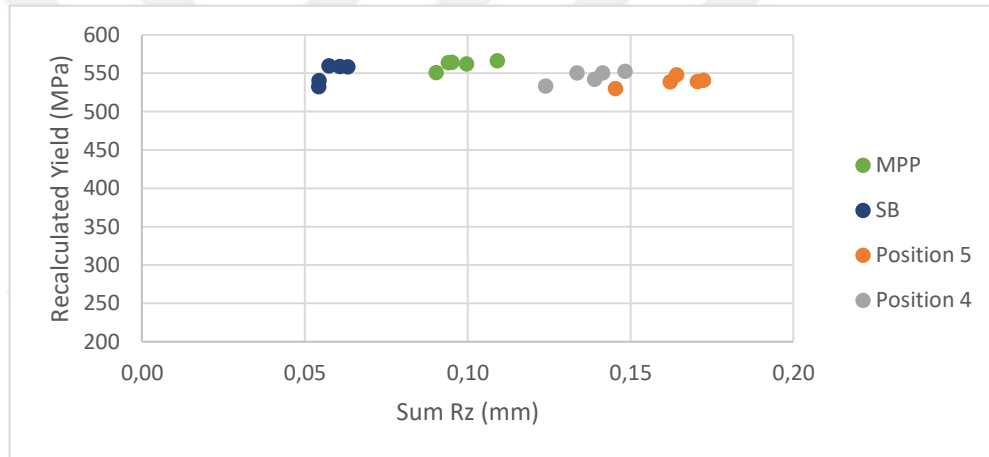


Figure 4.32 : Tensile test yield strength results with recalculated max stress for MPP, SB and AB samples.

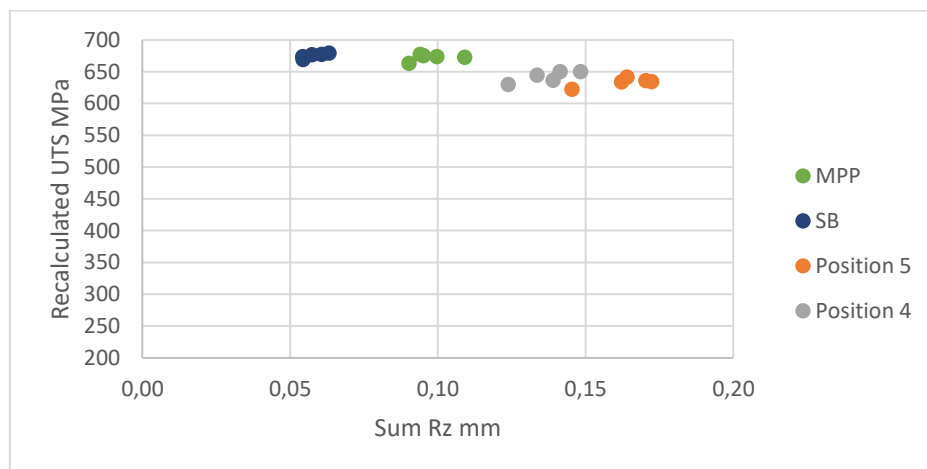


Figure 4.33 : Tensile test UTS results with recalculated max stress for MPP, SB and AB samples.

The average yield strength value for MPP samples (561 MPa) is lower than the average yield for SB samples (550 MPa) which might also be due to relatively lower roughness of SB samples. The percentage differences between MPP and AB is calculated as 4% and it is calculated as 1% for SB and AB samples.

4.4.2 Recalculated fatigue test results

4.4.2.1 As-built samples

As listed in Table 4.21, t-Rz values are calculated for the samples and inverse calculations are made; Applied loads are divided by the ‘roughness free thickness’ (t-Rz) values and max stress values are recalculated as shown in Figure 4.34.

Table 4.21 : t-Rt and t-Rz values calculated for AB fatigue samples of position 1 and 5 to obtain avg theoretical ‘roughness free thickness’. Delta displays the difference between measured thickness and ‘roughness free thickness’.

Status	Test	Position	Avg t mm	Avg t-Rz mm	Δ
AB	F	1	0,909	0,797	0,11
AB	F	5	0,983	0,810	0,17

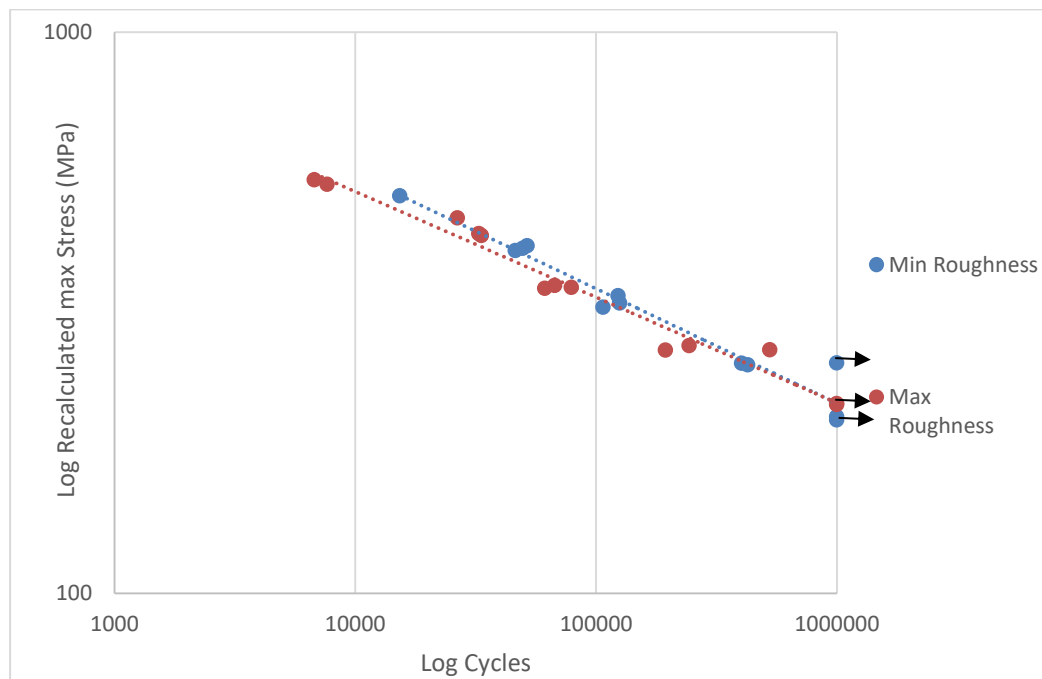


Figure 4.34 : Fatigue test results (Log-Log) with recalculated max stress for 26 AB samples tested at 5 stress levels for two different positions. Minimum roughness refers to position 1 and maximum roughness refers to position 5.

When recalculated max stress values are compared, curves for both minimum and maximum roughness values seem to match. It is seen that minimum surface roughness samples are subjected to 510 MPa stress while maximum roughness samples are subjected to 545 MPa instead of 450 MPa. The roughness free thickness for both maximum and minimum roughness samples are 0,8 mm. Therefore, the real cross section that the load is applied (smaller cross section area across the sample, see Figure 4.24) is same for both groups while higher load is applied onto the maximum roughness samples because of roughness effect on measured thickness. Consequently, the shift on Figure 4.20 showing raw fatigue test results can be explained by the thickness measurement difference rather than a mechanical behavior difference between minimum and maximum roughness.

4.4.2.2 Post-processed samples

Then the ‘roughness free thickness’ calculations are made for MPP and SB samples as shown in Table 4.22 and recalculated stress values are shown in Figure 4.35.

Table 4.22 : t-Rt and t-Rz values calculated for AB, MPP and SB fatigue samples of position 5 to obtain avg theoretical ‘roughness free thickness’. Delta displays the difference between measured thickness and ‘roughness free thickness’.

Status	Test	Position	t mm	t-Rz mm	Δ
MPP	F	5	0,882	0,786	0,09
SB	F	4	0,866	0,792	0,07
AB	F	5	0,983	0,810	0,17

As can be seen in Figure 4.35, even the recalculation is done, the shift to the right for the PP sample results is still significant.

Since, the fatigue results of AB samples for different locations are compared in the previous section, and there is no difference regarding the different surface roughness values, this shift can not be explained with the roughness difference between AB and PP samples. However, both SB and MPP samples are subjected to SB. Therefore, SB may have caused a compressive residual stress on PP samples which results in improved fatigue life. The time of sandblasting applied to SB and MPP samples is same but MPP still shows a better fatigue behavior. This can be explained by the increased hardness of MPP samples due to the anodization step in MPP after SB step.

Anodization creates oxide layer on the surface which has higher hardness and acts as an obstacle for crack initiation [52].

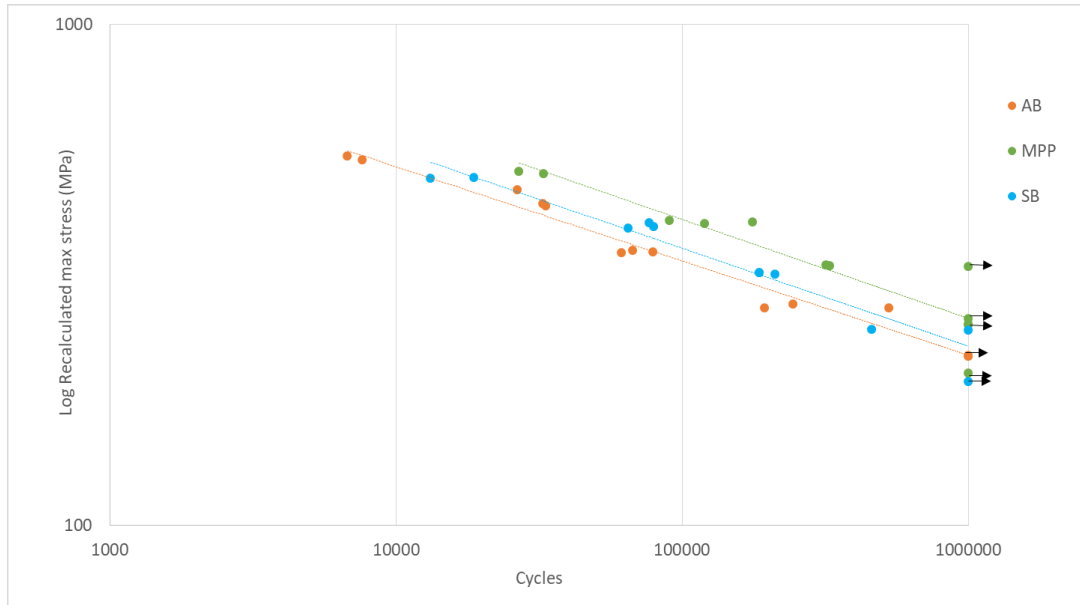


Figure 4.35 : Fatigue test results with recalculated max stress (log Recalculated max stress v. log Cycles) for 39 samples tested at 5 stress levels for AB, MPP and SB samples.

In summary, SB and MPP samples have better fatigue behavior than AB samples due to the compressive residual stress caused by SB. Also, for MPP samples, it is seen that even with the higher surface roughness, they have longer fatigue life than SB samples which can be explained by higher hardness caused by oxide layer.

5. CONCLUSIONS AND FUTURE WORK

In this work, regarding the different surface roughness observed along the opposite sides of products and across the build chamber, investigation of the surface roughness effect on mechanical behavior of SLM produced cpTi was carried out in order to detect possible negative effect on product quality.

Firstly, CM, tactile profilometer and SEM are compared as possible roughness measurement methods and it is shown that SEM is not an applicable method due to the incapability of determining high roughness values while CM and tactile profilometer are capable of quantifying issued surface roughness values. Considering the more practical application, low cost and adequacy for regular roughness range of finalized products, tactile profilometer is selected as the ideal roughness measurement method for this study.

Secondly, surface roughness across the build plate is quantified and roughness ranges are assigned for specific positions. As the most explicit conclusions can be expressed as the increased surface roughness for the right side and especially the bottom of the build chamber and for the orientations perpendicular to the laser beam.

From the optimization point of view, the process parameters can be examined in detail to determine the cause of evident difference between left and right side of the build chamber. To be able to use the build chamber more effectively, besides examination of left and right side difference, the effect of build height on part properties can be studied.

Also, as a result of proved relation between R_z and thickness, a new technique is suggested for the evaluation of tensile and fatigue test results. An equation to remove the effect of roughness on thickness is defined and 'roughness free thickness' is calculated to reassess the tensile and fatigue test results.

Recalculated tensile results with the eliminated roughness effect have shown clear consistency for all AB samples, which suggests that surface roughness has no effect on tensile strength, it has just an effect on measured cross section which is the result

of micrometer measurement limitation. For PP samples, there is still a small difference observed even after the recalculation is applied which may be the effect of residual stresses.

Also, when recalculation is applied on the fatigue test results, it is seen that difference appearing for varying surface roughness values are the result of thickness measurements affected by surface roughness and there is no significant effect of surface roughness on fatigue life of the samples.

However, for the post-processed samples, above conclusion can not be stated as clear. Since PP samples show different behavior, more in depth investigation should be made about PP samples.

One of the possible explanation for different behavior of PP samples might be the sand-blasting part of applied post-processing, since SB is a possible residual stress source. Also, hard oxide layer introduced by anodization can explain higher mechanical test results for MPP samples.

Sand-blasting effect on surface roughness and residual stress could be determined by differentiating sand-blasting time and applying mechanical tests.

Further, even surface roughness does not have an effect on mechanical behavior, it is still considered as a problem for asthetic concerns. Therefore, surface roughness difference across the build plate could be neutralized by determining the optimal post processing steps for the different roughness ranges.

REFERENCES

- [1] **Townsend, A., Senin, N., Blunt, L., Leach, R., & Taylor, J.** (2016). Surface texture metrology for metal additive manufacturing: a review. *Precision Engineering*, 46, 34-47. doi:10.1016/j.precisioneng.2016.06.001
- [2] **Dadbakhsh, S., Vrancken, B., Kruth, J., Luyten, J., & Humbeeck, J. V.** (2016). Texture and anisotropy in selective laser melting of NiTi alloy. *Materials Science and Engineering: A*, 650, 225-232. doi:10.1016/j.msea.2015.10.032
- [3] **Mumtaz, K., & Hopkinson, N.** (2009). Top surface and side roughness of Inconel 625 parts processed using selective laser melting. *Rapid Prototyping Journal*, 15(2), 96-103. doi:10.1108/13552540910943397
- [4] **Pyka, G., Kerckhofs, G., Papantoniou, I., Speirs, M., Schrooten, J., & Wevers, M.** (2013). Surface Roughness and Morphology Customization of Additive Manufactured Open Porous Ti6Al4V Structures. *Materials*, 6(10), 4737-4757. doi:10.3390/ma6104737
- [5] **Kleszczynski, S., Ladewig, A., Friedberger, K., Zur Jacobsmühlen, J., Merhof, D., & Witt, G.** (2015). Position dependency of surface roughness in parts from laser beam melting systems. In *Solid Freeform Fabrication Symposium*, Austin, TX.
- [6] **Shemtov-Yona, K., & Rittel, D.** (2015). An Overview of the Mechanical Integrity of Dental Implants. *BioMed Research International*, 2015, 1-11. doi:10.1155/2015/547384
- [7] **Suh, C. H., Jung, Y., & Kim, Y. S.** (2010). Effects of thickness and surface roughness on mechanical properties of aluminum sheets. *Journal of Mechanical Science and Technology*, 24(10), 2091-2098. doi:10.1007/s12206-010-0707-7
- [8] **Hurtado, O. J., Tiralongo, V. C., & Aguirre, M. C.** (2017). Effect of surface hardness and roughness produced by turning on the torsion mechanical properties of annealed AISI 1020 steel. *Revista Facultad de Ingeniería Universidad de Antioquia*, (84), 55-59. doi:10.17533/udea.redin.n84a07
- [9] **Whitehouse, D. J.** (2003). *Handbook of Surface and Nanometrology*, 38. doi:10.1887/0750305835
- [10] **Bhushan, B.** (2013). *Introduction to Tribology*. doi:10.1002/9781118403259
- [11] **Vörös, J., Wieland, M., Ruiz-Taylor, L., Textor, M., & Brunette, D. M.** (2001). Characterization of Titanium Surfaces. *Engineering Materials Titanium in Medicine*, 87-144. doi:10.1007/978-3-642-56486-4_5

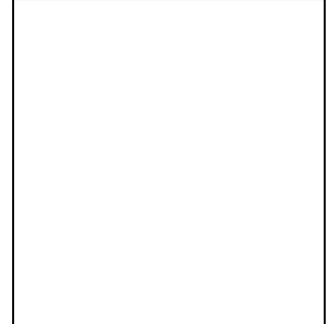
- [12] **Thompson, A., Maskery, I., & Leach, R. K.** (2016). X-ray computed tomography for additive manufacturing: a review. *Meas. Sci. Technol.*, 27(7), 072001.
- [13] **Thompson, A., Senin, N., Giusca, C., & Leach, R.** (2017). Topography of selectively laser melted surfaces: A comparison of different measurement methods. *CIRP Annals*, 66(1), 543-546. doi:10.1016/j.cirp.2017.04.075
- [14] **Bremen, S., Meiners, W. and Diatlov, A.** (2012), Selective Laser Melting. *LTJ*, 9: 33–38. doi:10.1002/latj.201290018
- [15] **European Powder Metallurgy Association.** (2015). [Brochure]. Author. Retrieved from <https://www.epma.com/additive-manufacturing>
- [16] **Attar, H., Calin, M., Zhang, L. C., Scudino, S., & Eckert, J.** (2014). Manufacture by selective laser melting and mechanical behavior of commercially pure titanium. *Materials Science and Engineering: A*, 593, 170-177.
- [17] **Wysocki, B., Maj, P., Krawczyńska, A., Roźniatowski, K., Zdunek, J., Kurzydłowski, K. J., & Świąszkowski, W.** (2017). Microstructure and mechanical properties investigation of CP titanium processed by selective laser melting (SLM). *Journal of Materials Processing Technology*, 241, 13-23. doi:10.1016/j.jmatprotec.2016.10.022
- [18] **Yadroitsev, I., & Smurov, I.** (2011). Surface morphology in selective laser melting of metal powders. *Physics Procedia*, 12, 264-270.
- [19] **Kwasniak, P., Muzyk, M., Garbacz, H., & Kurzydłowski, K.J.** (Jan 2014). Influence of oxygen content on the mechanical properties of hexagonal Ti—First principles calculations. *Materials Science and Engineering A, Structural Materials: Properties, Microstructure and Processing*, 74-79. doi:10.1016/j.msea.2013.10.004
- [20] **Osakada, K., & Shiomi, M.** (2006). Flexible manufacturing of metallic products by selective laser melting of powder. *International Journal of Machine Tools and Manufacture*, 46(11), 1188-1193.
- [21] **Srivatsan, T. S., & Sudarshan, T. S. (Eds.).** (2015). *Additive manufacturing: innovations, advances, and applications*. CRC Press.
- [22] **Kruth, J. P., Badrossamay, M., Yasa, E., Deckers, J., Thijs, L., & Van Humbeeck, J.** (2010, April). Part and material properties in selective laser melting of metals. In *Proceedings of the 16th international symposium on electromachining*.
- [23] **Thijs, L., Verhaeghe, F., Craeghs, T., Van Humbeeck, J., & Kruth, J. P.** (2010). A study of the microstructural evolution during selective laser melting of Ti–6Al–4V. *Acta Materialia*, 58(9), 3303-3312.
- [24] **Yadroitsev I, Pavlov M, Bertrand Ph, Smurov I.** (2009). Mechanical properties of samples fabricated by selective laser melting. In: *14e`mes Assises Europe`ennes du Prototypages & Fabrication Rapide*, Paris;.
- [25] **Hitzler, L., Hirsch, J., Merkel, M., Hall, W., & Öchsner, A.** (2017). Position dependent surface quality in selective laser

melting. *Materialwissenschaft Und Werkstofftechnik*, 48(5), 327-334.
doi:10.1002/mawe.201600742

- [26] **Razvan, P., & Ancuta, P.** (2016). Applications of the Selective Laser Melting Technology in the Industrial and Medical Fields. In *New Trends in 3D Printing*. InTech.
- [27] **Yap, C. Y., Chua, C. K., Dong, Z. L., Liu, Z. H., Zhang, D. Q., Loh, L. E., & Sing, S. L.** (2015). Review of selective laser melting: Materials and applications. *Applied physics reviews*, 2(4), 041101.
- [28] **Lucideon.** (n.d.). Additive Manufacturing – A Metallurgical Perspective [Brochure]. Author. Retrieved from www.lucideon.com
- [29] **Alla, R. K., Ginjupalli, K., Upadhya, N., Shammash, M., Ravi, R. K., & Sekhar, R.** (2011). Surface roughness of implants: a review. *Trends in Biomaterials and Artificial Organs*, 25(3), 112-118.
- [30] **Kruth, J. P., Yasa, E., & Deckers, J.** (2008, September). Roughness improvement in selective laser melting. In *Proceedings of the 3rd international conference on polymers and moulds innovations* (pp. 170-183).
- [31] **Mertens, R., Clijsters, S., Kempen, K., & Kruth, J. P.** (2014). Optimization of scan strategies in selective laser melting of aluminum parts with downfacing areas. *Journal of Manufacturing Science and Engineering*, 136(6), 061012.
- [32] **Vaithilingam, J., Prina, E., Goodridge, R. D., Hague, R. J., Edmondson, S., Rose, F. R., & Christie, S. D.** (2016). Surface chemistry of Ti6Al4V components fabricated using selective laser melting for biomedical applications. *Materials Science and Engineering: C*, 67, 294-303
- [33] **Sul, Y. T., Johansson, C. B., Petronis, S., Krozer, A., Jeong, Y., Wennerberg, A., & Albrektsson, T.** (2002). Characteristics of the surface oxides on turned and electrochemically oxidized pure titanium implants up to dielectric breakdown: the oxide thickness, micropore configurations, surface roughness, crystal structure and chemical composition. *Biomaterials*, 23(2), 491-501.
- [34] **Strano, G., Hao, L., Everson, R. M., & Evans, K. E.** (2013). Surface roughness analysis, modelling and prediction in selective laser melting. *Journal of Materials Processing Technology*, 213(4), 589-597.
- [35] **Dezso, G., & Kósa, P.** (2012). Roughness of plane faces produced by additive manufacturing. *Annals of the Faculty of Engineering Hunedoara*, 10(2), 181.
- [36] **Badiru, A. B., Valencia, V. V., & Liu, D. (Eds.).** (2017). *Additive Manufacturing Handbook: Product Development for the Defense Industry*. CRC Press
- [37] **Triantaphyllou, A., Giusca, C. L., Macaulay, G. D., Roerig, F., Hoebel, M., Leach, R. K., ... & Milne, K. A.** (2015). Surface texture measurement for additive manufacturing. *Surface Topography: Metrology and Properties*, 3(2), 024002.

- [38] **Whitehouse, D. J.** (1994). Handbook of surface metrology. CRC Press.
- [39] **Vandenbroucke, B., & Kruth, J. P.** (2007). Selective laser melting of biocompatible metals for rapid manufacturing of medical parts. *Rapid Prototyping Journal*, 13(4), 196-203.
- [40] **Parithimarkalaignan, S., & Padmanabhan, T. V.** (2013). Osseointegration: an update. *The Journal of Indian Prosthodontic Society*, 13(1), 2-6.
- [41] **Guilherme, A. S., Henriques, G. E., Zavanelli, R. A., & Mesquita, M. F.** (2005). Surface roughness and fatigue performance of commercially pure titanium and Ti-6Al-4V alloy after different polishing protocols. *The Journal of Prosthetic Dentistry*, 93(4), 378-385. doi:10.1016/j.prosdent.2005.01.010
- [42] **Haitjema H.** (2014) Roughness. In: *The International Academy for Production Engineering*, Laperrière L., Reinhart G. (eds) CIRP Encyclopedia of Production Engineering. Springer, Berlin, Heidelberg
- [43] **Bhushan, B.** (2001). Modern tribology handbook, Volume 1. CRC press.
- [44] **Hobson, T., & PRECISION'S, G. U. I. D. E.** (2003). Exploring Surface Texture: A fundamental guide to the measurement of surface finish. Leicester–Inglaterra, 4th edition, ed. Taylor Hobson Ltda, 100.
- [45] **Thomas, T. R.** (1999). Rough surfaces
- [46] **Rosén, S., Thomas, T. R., & Rosén, B. G.** (2013). The Stedman diagram revisited. *Surface Topography: Metrology and Properties*, 2(1), 014005.
- [47] **Olympus.** (n.d.). Introduction to Surface Roughness Measurement [Brochure]. Author. Retrieved from <https://www.olympus-ims.com/en/knowledge/metrology/roughness/>
- [48] **Gadelmawla, E. S., Koura, M. M., Maksoud, T. M. A., Elewa, I. M., & Soliman, H. H.** (2002). Roughness parameters. *Journal of materials processing technology*, 123(1), 133-145.
- [49] **Leach, R. (Ed.).** (2011). Optical measurement of surface topography (Vol. 14, p. 326). Berlin: Springer.
- [50] **Kohles, S. S., Clark, M. B., Brown, C. A., & Kenealy, J. N.** (2004). Direct assessment of profilometric roughness variability from typical implant surface types. *International Journal of Oral & Maxillofacial Implants*, 19(4).
- [51] **Librera, E., Riva, G., Safarzadeh, H., & Previtali, B.** (2015). On the use of Areal Roughness Parameters to Assess Surface Quality in Laser Cutting of Stainless Steel with CO2 and Fiber Sources. *Procedia CIRP*, 33, 532-537. doi:10.1016/j.procir.2015.06.069
- [52] **Niinomi, M., & Akahori, T.** (2010). Improvement of the fatigue life of titanium alloys for biomedical devices through microstructural control. *Expert Review of Medical Devices*, 7(4), 481-488. doi:10.1586/erd.10.16

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- **Tanyeri B., Akdas G., Senol S., Akyil C., Urgen M.** 2017. Role of Intermetallic Size and Distribution on the Anodizing Quality of 1xxx and 6xxx Series Aluminum Alloys. Aluminum Anodizing & Extrusion Summit. Aluminum Anodizers Council. US.