

ISTANBUL TECHNICAL UNIVERSITY★ GRADUATE SCHOOL

**COMPARISON OF BIOACTIVITIES OF Ti6Al4V IMPLANTS AFTER
MULTIPLE SURFACE TREATMENTS**



M.Sc. THESIS

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Department of Nano Science& Nano Engineering

Nano Science& Nano Engineering Programme

FEBRUARY 2025

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

**ÇOKLU YÜZEY İŞLEMLERİ UYGULANAN Ti6Al4V İMPLANTLARIN
BİYOAKTİVİTELERİNİN KARŞILAŞTIRILMASI**

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Date of Defense : 13 February 2025



FOREWORD

I would like to express my gratitude to my advisor Prof. Dr. Nilgün BAYDOĞAN for her wisdom and guidance during my thesis preparation. I would like to thank the ITU BAP unit (44208) for supporting the financing of my thesis.

I would like to thank my mom, dad and my cousin Mehmet ANIL, who have always been with me for all their support during this period. I am so glad i have you. I would like to express my sincere thanks to Asst. Prof. Dr. Faiz MUHAFFEL who guided my thesis. I would like to thank dear Ecem ERMAN who answered all my questions patiently, supported and guided me.

January 2025

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ABBREVIATIONS

Al₂O₃	: Aluminium oxide
BCC	: Body-Centered Cubic
CaP	: Calcium Phosphate
CBCT	: Cone Beam Computed Tomography
cpTi	: Commercially Pure Titanium
CT	: Computed Tomography
ECM	: Extracellular Matrix
EDS	: Energy Dispersive Spectrometer
FTIR	: Fourier transform infrared spectroscopy
H₂O₂	: Hydrogen peroxide
HA	: Hydroxyapatite
HCP	: Hexagonal Close-Packed
MAO	: Micro-Arc Oxidation
MRI	: Magnetic Resonance Imaging
PEEK	: Polyether ether ketone
PEG	: Polyethylene glycol
PMMA	: Poly (methyl methacrylate)
SBF	: Simulated Body Fluid
SEM	: Scanning Electron Microscope
SS	: Stainles Steel
Ti₆Al₄V	: Titanium Grade 5
TiO₂	: Titanium dioxide
TPS	: Titanium Plasma Spray
VEGF	: Vascular Endothelial Growth Factor
wt.	: Weight
XRD	: X-ray Diffraction



SYMBOLS

Ti	: Titanium
C	: Carbon
H	: Hydrogen
Ta	: Tantalum
Al	: Aluminum
N	: Nitrogen
O	: Oxygen
Mn	: Manganese
V	: Vanadium
Cr	: Chromium
Fe	: Iron
P	: Phosphorus
α	: Alpha
β	: Beta



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COMPARISON OF BIOACTIVITIES OF Ti6Al4V IMPLANTS AFTER MULTIPLE SURFACE TREATMENTS

SUMMARY

Teeth are very important organs in terms of completing digestion, protecting organs and having an aesthetic appearance, and they can be lost due to reasons such as incorrect applications, poor oral hygiene and various disorders. Although many applications have been sought throughout history to solve tooth loss, the most effective method is dental implant applications. Titanium and its alloys are frequently preferred in biomedical applications due to their good mechanical properties, high biocompatibility and corrosion resistance. Osseointegration is very important for the success of the implant in dental implant applications using pure titanium and its alloys. Osseointegration can be defined as the adaptation of the dental implant with the lower jaw bone (mandible) or upper jaw bone (maxilla) and its wrapping by bone tissue. There are many factors in providing osseointegration in dental implants, one of which is the surface properties of the implant. Osseointegration can be increased and accelerated by changes made in the surface properties, thus shortening the treatment period. The main purpose of surface modifications in dental implants is to increase osseointegration by making changes in the surface morphology and chemistry of the implant material. One of the surface modification methods, SLA (sandblasting, acid etching), is an effective surface treatment in dental implant applications that increases surface roughness and thus osseointegration by creating a larger bone-implant contact area. This thesis study aims to investigate the effect of the SLA surface modification process, which is frequently used in the field of dental implants, on the surface morphology of Grade 5 titanium implants and disc samples using different grit sizes, by examining various fallen implant surfaces. It was thought that the surface properties of the material could be damaged as a result of the SLA process. For this reason, implants roughened with the SLA method were examined and it was investigated why they could not provide osseointegration. First, SEM (Scanning Electron Microscope) images of these implants were taken, then radiological images were taken to determine whether there were structures such as fractures and cracks. In the examination of SEM analyzes, it was observed that there were various deteriorations, micro-fracture-like structures, inhomogeneous distributions and deformations on their surfaces and implant structures. Later, it was understood from the radiographic images obtained using X-rays that there were no fractures in their internal structures. Then, in order to investigate the effect of different grit sizes and surface roughness on osseointegration, disc and implant samples were used to perform SLA surface modification. In the first stage, SEM data of the sample without SLA process was taken, images were compared after sandblasting and it was understood that sandblasting and acidizing processes were successful. Sandblasting sands of various sizes were used to better understand the effect of surface roughness resulting from sandblasting and acid etching on osseointegration.

Aluminum oxide sands of 78 nm, 180 nm, 3-19 μm , 63-90 μm , 90-125 μm , 150-212 μm , 212-300 μm sizes were used. During the sandblasting process, it was observed that sands of 78 nm and 180 nm sizes were not suitable for sandblasting and could not provide roughness. Sandblasting was successfully carried out with sand grains of other sizes. After the sandblasting process, the samples were washed with pure water in an ultrasonic washing machine to remove contaminants from the surface and prepare them for the etching process. Citric acid, oxalic acid and binary combinations of these acids were applied to the implant and disc samples and acid etched on the sandblasted surface. EDS-SEM images of the samples were taken after the SLA surface treatment. In the SEM images taken, it was seen that higher roughness was obtained on the surfaces of the implants and discs compared to the surfaces without sandblasting and acid etching. The rough surfaces obtained as a result of the process can be seen from the contrasts and field depths in the SEM images and show that the process was successfully performed. It was observed that the field depth decreased as the grit sizes decreased. Then, the surface roughnesses of the samples were measured and compared. The lowest surface roughness was obtained in 3-19 μm aluminum oxide grit and binary acid, while the highest roughness was obtained in 90-125 μm grit and oxalic acid. Then, Simulated Body Fluid (SBF) was prepared to test the bioactivity of the disc and implant samples. SBF is an artificial fluid that mimics body fluids such as natural blood plasma. This fluid is used to test how the used material will react in a biological environment. SBF contains ionic compounds such as Sodium, Calcium, Potassium, Magnesium and Phosphate, which are also found in blood plasma. There is a correlation between in vivo bone growth and in vitro apatite formation ability. Therefore, in order to compare apatite formation mechanisms, disk samples and implants were immersed in SBF and examined in terms of apatite formation mechanism. While preparing the simulated body fluid, weighings were performed on a high-resolution precision balance, and since temperature changes may cause precipitation in the solution, the temperature was kept constant. SEM images were taken after the samples were kept in the prepared SBF for 14 days. No significant apatite presence was observed in the SEM images. Micro arc oxidation (MAO) method was used to increase bioactivity on the surface. Then, the samples were washed appropriately and subjected to MAO treatment, which is an electrochemical modification process that uses the principle of oxidation of the material surface using high-energy arcs. The purpose of this surface treatment is to improve the biological properties of the implants. After the MAO treatment, the samples were washed again with pure water and dried. After the MAO treatment, the sample was immersed in SBF again. It was kept in SBF again for 14 days. After the samples were removed, they were washed with distilled water and stored appropriately, and then SEM analyses were performed again. Apatite formation was observed on the surfaces of the samples treated with MAO in SEM images and confirmed in EDS analysis. XRD and FTIR analyses were then performed to comment on the apatite structures of the samples. The results obtained in these analyses showed that the sand sizes that provided optimum roughness and best supported apatite formation were F80 and F120 (90-125 μm , 150-212 μm) samples.

ÇOKLU YÜZEY İŞLEMLERİ UYGULANAN Ti6Al4V İMPLANTLARIN BİYOAKTİVİTELERİNİN KARŞILAŞTIRILMASI

ÖZET

Dişler sindirimin tamamlanması, organların korunması ve estetik bir görünüm açısından oldukça önemli bir organ olup yanlış uygulamalar, kötü ağız hijyeni, kaza veya yaralanmalar, çeşitli rahatsızlıklar gibi sebeplerden kaybedilebilir. Tarih boyunca birçok uygulama ile diş kaybına çözümü aranmış olsa da en etkili yöntem diş implantı uygulamalarıdır. Diş implantları, çeşitli nedenlerle kaybedilen dişlerin yerine yerleştirilen yapay diş kökleridir. İmplantlar, çene kemiğine yerleştirilen vida şeklinde yapılar olup, üzerine protez malzemesi uygulanarak dişlerin fonksiyonları yerine getirilmeye çalışılır. Farklı implant türleri, kullanılan malzemeler ve yerleştirilme yöntemlerine göre sınıflandırılır. Endosteal İmplantlar, en yaygın kullanılan diş implant türüdür ve çene kemiğine doğrudan yerleştirilir. Genellikle titanyumdan üretilen bu implantlar, diş kökünü taklit ederek yerleşim sonrası zamanla kemikle bağlantı oluşturur ve osseointegrasyon sağlar, bu da implantın sağlam bir şekilde yerinde kalmasını sağlar. Endosteal implantlar, tek bir diş kaybından büyük köprü ve protez uygulamalarına kadar geniş bir kullanım alanına sahiptir. Bu implant türü, sağlam çene kemiğine sahip bireylerde başarılı sonuçlar verir. İmplantın üzerine diş protezi yerleştirildikten sonra, doğal diş gibi fonksiyon görmeye başlar. Biyomedikal uygulamalarda titanyum ve alaşımları sahip oldukları iyi mekanik özellikler, yüksek biyouyumluluk ve korozyon direnci gibi özellikleri nedeniyle sıklıkla tercih edilmektedir. Saf titanyum ve alaşımları kullanılarak yapılan dental implant uygulamalarında implantın başarısı bakımından osseointegrasyon çok önemlidir. Osseointegrasyon, dental implantın altçene kemiği (mandibula) veya üst çene kemiği (maksilla) ile uyum sağlanması ve kemik dokusu tarafından sarılması olarak tanımlanabilir. Osseointegrasyon, diş implantının çene kemiğiyle kaynaşma süreci anlamına geldiğinden implant başarısı açısından çok önemlidir ve birçok faktörden etkilenir. Kemiğin kalitesi ve miktarı, implantın malzemesi, yüzey özellikleri, cerrahi teknik ve uygulama, hastanın yaşı ve genel sağlık durumu bu süreci doğrudan etkiler. İyi kalitede kemik ve biyouyumlu malzemeler, osseointegrasyonu hızlandırırken, pürüzlü implant yüzeyleri kemik hücrelerinin tutunmasını destekler. Sigara kullanımı, kan akışını engelleyerek iyileşmeyi geciktirebilir ve enfeksiyon riskini artırabilir. Ayrıca, implantın üzerine diş protezinin ne zaman yerleştirileceği ve uygulanan yükler de entegrasyon sürecini etkiler; erken yükleme ve uygulanan yüksek kuvvetler osseointegrasyonu zorlaştırabilir. Bu faktörlerin her biri, implantın başarısını ve uzun vadeli sağlığını belirler, bu yüzden tedavi süreci dikkatlice planlanmalıdır ve seçim hasta profiline ve hastanın sağlık geçmişine uygun yapılmalıdır. Osseointegrasyonu etkileyen bir diğer faktör ise implantın yüzey özellikleridir. Yüzey özelliklerinde yapılan değişikliklerle osseointegrasyon artırılabilir ve hızlandırılabilir, dolayısıyla tedavi süresi kısaltılabilir. Dental implantlarda yüzey modifikasyonlarının temel amacı, implant malzemesinin yüzey morfolojisinde ve kimyasında değişiklikler yaparak osseointegrasyonu arttırmaktır. Yüzey modifikasyon yöntemlerinden biri olan

SLA (kumlama, asitle aşındırma) daha büyük kemik-implant temas alanı oluşturarak yüzey pürüzlülüğünü ve böylelikle osseointegrasyonu arttıran dental implant uygulamalarında etkin bir yüzey işlemidir. Bu tez çalışması, dental implant alanında sıklıkla kullanılan, SLA yüzey modifikasyon işlemini, farklı kum boyları kullanılarak Grade 5 titanyum implantın ve disk numunelerinin yüzey morfolojisine olan etkisini, çeşitli düşen implant yüzeylerinin incelenmesinden yola çıkarak hedeflemektedir. SLA işlemi sonucunda malzemenin yüzey özelliklerinde tahribat meydana gelebileceği düşünülmüştür. Bu nedenle; SLA yöntemi ile pürüzlendirilmiş, ağıza yerleştirilmiş fakat başarı sağlayamayıp düşen implantlar incelenerek neden osseointegrasyon sağlayamadığı araştırılmıştır. Önce SEM (Taramalı Elektron Mikroskobu) görüntüleri alınarak yüzey yapıları incelenen bu implantların daha sonra kırık ve çatlak gibi yapılarının var olup olmadığının tespiti için üç boyutlu görüntülerinin incelenmesi için radyolojik görüntüleri alınmıştır. SEM analizlerinin incelenmesinde, yüzeylerinde ve implant yapılarında çeşitli bozulmalar, mikro-kırık benzeri yapılar, yüzeylerinde homojen olmayan dağılımlar ve deformasyonlar olduğu görülmüştür. Daha sonra X ışınları kullanılarak elde edilen radyografi görüntülerinde iç yapılarında kırık olmadığı anlaşılmıştır. Bu aşamalardan sonra farklı kum boyları ve yüzey pürüzlülüğünün osseointegrasyona olan etkisini araştırmak amacıyla SLA yüzey modifikasyonu gerçekleştirmek için disk ve implant numuneleri hazırlanmıştır. İmplantlar temin edilirken, disk numuneler titanyum çubuktan kesilmiştir. İlk aşamada SLA işlemi gerçekleştirilmemiş numunenin SEM analizlerinin alınması, kumlama sonrasında görüntülerin karşılaştırılması sonucu kumlama ve asitleme işleminin başarıyla sonuçlandığı anlaşılmıştır. Kumlama ve asitleme sonucu oluşan yüzey pürüzlülüğünün osseointegrasyona etkisinin daha iyi anlaşılabilmesi için çeşitli boyalarda kumlama kumları kullanılmıştır. 78 nm, 180 nm, 3-19 µm, 63-90 µm, 90-125 µm, 150-212 µm, 212-300 µm boyutlarındaki Alüminyum oksit kumları kullanılmıştır. Kumlama işlemi sırasında 78 nm ve 180 nm boyutlarındaki kumların kumlama için uygun olmadığı ve pürüzlülük sağlayamadığı görülmüştür. Bu kumlar kumlama cihazı içinde tıkanmış ve disk yüzeylerinde herhangi bir pürüzlülük sağlamamışlardır. Diğer boyutlardaki kum tanecikleri ile kumlama başarıyla gerçekleştirilmiştir. Kumlama işleminin ardından yüzeydeki kirleticilerden arındırmak ve etching işlemine hazırlamak için numuneler ultrasonik yıkama makinesinde saf su ile yıkanmıştır. İmplant ve disk numunelerine sitrik asit, oksalik asit ve bu asitlerin ikili kombinasyonları uygulanarak kumlanan yüzey üzerine asitle dağlama yapılmıştır. SLA yüzey işleminin ardından numunelerin EDS-SEM görüntüleri alınmıştır. Alınan SEM görüntülerinde implant ve disklerin yüzeylerinde kumlama ve asitle dağlama yapılmayan yüzeye göre daha yüksek pürüzlülük elde edildiği görülmüştür. İşlem sonucunda elde edilen pürüzlü yüzeyler SEM görüntülerindeki kontrastlardan ve alan derinliklerinden görülebilmekte ve işlemin başarılı bir şekilde gerçekleştirildiğini göstermektedir. Kum boyutları azaldıkça alan derinliğinin azaldığı gözlemlenmiştir. Daha sonra numunelerin yüzey pürüzlülükleri ölçülerek karşılaştırılmıştır. En düşük yüzey pürüzlülüğü 3-19 µm alüminyum oksit kum ve ikili asitte elde edilirken, en yüksek pürüzlülük 90-125 µm kum ve oksalik asitte elde edilmiştir. Daha sonra disk ve implant numunelerinin biyoaktivitesini test etmek için Simüle Edilmiş Vücut Sıvısı (SBF) hazırlanmıştır. SBF, doğal kan plazması gibi vücut sıvılarını taklit eden yapay bir sıvıdır. Bu sıvı, kullanılan malzemenin biyolojik ortamda nasıl tepki vereceğini test etmek için kullanılır. SBF, kan plazmasında da bulunan Sodyum, Kalsiyum, Potasyum, Magnezyum ve Fosfat gibi iyonik bileşikler içerir. İn vivo kemik büyümesi ile in vitro apatit oluşum yeteneği arasında bir korelasyon vardır. Bu nedenle apatit oluşum mekanizmalarını karşılaştırmak amacıyla disk numuneleri ve implantlar

SBF'ye daldırılarak apatit oluşum mekanizması açısından incelenmiştir. Simüle edilmiş vücut sıvısı hazırlanırken tartımlar yüksek çözünürlüklü hassas terazide gerçekleştirilmiş ve numune sırası önemli olduğundan standartta yazıldığı sırayla eklenmiştir. Devamlı olarak hem sıcaklık hem de pH ölçümü gerçekleştirilmiştir. Sıcaklık değişimleri çözeltide çökmeye neden olabileceğinden sıcaklık sabit tutulmuştur. SLA ile muamele edilen numuneler hazırlanan SBF'de 14 gün bekletilmiş ve ardından SEM görüntüleri alınmıştır. Elde edilen SEM görüntülerinde önemli bir apatit varlığı gözlenmemiştir. Yüzeyde biyoaktiviteyi arttırmak için Mikro ark oksidasyonu (MAO) metodu kullanılmıştır. Bu aşamada numuneler uygun yüksek enerjili arklar kullanılarak malzeme yüzeyinin oksitlenmesi prensibini kullanan bir elektrokimyasal modifikasyon işlemi olan MAO işlemine tabi tutulmuştur. Bu yüzey işleminin amacı implantların biyolojik özelliklerini iyileştirmektir. Bu kurulumda, elektrolit içeren paslanmaz çelik bir kap katot olarak ve numune anot olarak kullanılmıştır. MAO işlemi, toplam 2,5 litre çözeltide 20,5 g/L Kalsiyum Asetat ($C_4H_6CaO_4$) ve 14,4 g/L Monosodyum fosfat (NaH_2PO_4) kullanılarak gerçekleştirilmiştir. 300 V pozitif voltaj ve 60 V negatif voltaj uygulanan işlem 3 dakika sürmüştür. Elektrolit kabı, işlem sırasında elektrolit sıcaklığının 30 °C'nin altında kalmasını sağlamak için harici bir soğutma ünitesiyle soğutuldu. MAO işleminden sonra numuneler tekrar saf su ile yıkanmış ve oda sıcaklığında kurutulmuştur. MAO işleminden sonra numune tekrar SBF'ye daldırılmıştır. Tekrar 14 gün SBF'de tutuldu. Numuneler çıkarıldıktan sonra uygun şekilde saf su ile yıkanıp saklandıktan sonra SEM analizleri tekrar alındı. SEM görüntülerinde MAO ile işlem gören numunelerin yüzeylerinde apatit oluşumu gözlemlendi ve EDS analizinde doğrulandı. Literatürdeki diğer MAO yüzeyleri ve pikleri ile uyumlu olduğu görülmüştür. Daha sonra numunelerin apatit yapıları hakkında yorum yapabilmek için XRD ve FTIR analizleri yapıldı. Elde edilen XRD analizi verilerinde tüm numunelerde hidroksiapatit yapısı ve rutil TiO_2 yapılarının oluştuğu görülmüştür. En yüksek yoğunluktaki apatit oluşumu, F80 C numunesinde görülmüş ardından F120 numuneleri tarafından takip edilmiştir. Ardından FTIR analizi gerçekleştirilmiştir. FTIR analizi sonucu, F-80 ve F-120 parçacıklarıyla elde edilen özgül pürüzlülük, yüzey enerjisini ve aşındırma maddeleriyle ve sonraki işlemlerle etkileşimi artırmak için ideal olabileceği düşünülmüştür. Optimum büyüklükten daha büyük alüminyum oksit parçacıkları disk yüzeyinde derin ve belirgin pürüzlülükler oluşturmaktadır. Bu daha yüksek yüzey pürüzlülüğü daha az aktif kimyasal bölgeye ve dolayısıyla yüzey bileşikleriyle ilgili piklerin yoğunluğunda bir azalmaya yol açabilirken fazla küçük parçacıklar yüzeyi yeterince değiştirmek için yeterli enerji vermeyebilir. Orta boyut, alt tabakaya aşırı zarar vermeden etkili yüzey pürüzlendirmesine olanak tanır ve sonraki kimyasal reaksiyonlar için elverişli bir ortam yaratır. Bu analizlerde elde edilen sonuçlarda optimum pürüzlülük elde edilmesini sağlayan ve apatit oluşumunu en iyi destekleyen kum boylarının F80 ve F120 (90-125 μm , 150-212 μm) numuneleri olduğu görülmüştür.



1. INTRODUCTION

Teeth are vital organs that basically break down food into small pieces, allowing digestion to begin and interacting better with enzymes. For humans, teeth are crucial not only for the full start of digestive function, but also for speech, aesthetically good appearance and protection of organs [1]. Teeth, which are important in many respects, can be lost for various reasons. Reasons such as dental caries, (caused by poor oral hygiene, environmental chemicals, smoking, drugs, elemental toxic substances), wrong root canal treatment, injuries, diseases or habits of the patient, and gum diseases may cause tooth loss [2]. Dental health, which is also related to general health, is very important for holistic health [3]. Dental implants are regarded as the best solution for tooth loss. Dental implants are biocompatible materials placed in the jawbone to restore the patient's normal function, speech, health and aesthetics in cases where natural teeth are lost due to periodontal disease, failed root canal, injury or other reasons [3]. Dental implant applications are developing day by day [4].

1.1 History Of Dental Implants

Teeth are indispensable organs for the correct pronunciation of sounds and an aesthetic appearance. Throughout history, people have tried to replace missing teeth in order to restore the person's normal functions, to provide an facial aesthetic appearance or to show status. The history of artificial teeth applications that can replace teeth lost due to various reasons is very old [5].

Early dental practices give us information about both the beginnings of these practices that turned them into a science and the foundations on which modern dentistry is built [6]. Since the tooth is the best protected structure in the body, it has been helpful in gaining knowledge about dental practices.

It has been found that many dental practices were performed in ancient Egypt based on information obtained from various papyrus, mummies and remains.

Examination of the mummies revealed that animal teeth carved from ivory were used and this was one of the earliest examples of primitive implantology [7].

In the archaeological excavations of the Maya civilizations, it was observed that the missing teeth in the lower jaw were replaced by shell fragments in the 600s AD, and in the studies carried out in the following years, bone formation was provided around these structures [8].

In the 17th century in Europe, implant structures were used to replace the missing teeth obtained from some substances and animals, and in the 18th century, the missing teeth were tried to be filled by using teeth taken from donors but the success of these attempts is questionable [9]. One of the most important events that started the history of modern dental implants was the idea of using metals as prosthetics in dental restorations during the Second World War by Dr. Norman Goldberg [10]. In 1941, after the method of placing a metal structure with vertical extensions was described, inspired by this work, Dr. Norman Goldberg was founded in 1948 by Dr. Collaborating with Aaron Gershkoff, he came up with a new design. Later on, such designs were called subperiosteal implants [11]. One of the other most important developments in terms of modern implantology has been Branemark's studies that provided the definition of osseointegration. In his studies of bone, Branemark discovered that bone can grow around titanium metal without causing rejection. Branemark, who carried out many more studies afterward, named this phenomenon as osseointegration [12]. Osseointegration is defined as the direct structural and functional connection between implant and the bone [13]. Branemark, who carried out many experiments on humans and animals, successfully placed the first root form titanium dental implant in an edentulous patient who had tooth loss due to a jaw disorder in 1965. Biocompatibility and processability have been shown as the reason for the use of titanium implants [14]. Branemark has conducted many studies on the use of titanium due to its wonderful properties. He subsequently co-founded a company to develop and market implantology. In the following years, he published the results of his experiments, and many researchers were trained in the Branemark system and methods [15].

The use of titanium dental implants was approved by the FDA in 1982. In 1983, computer-aided design and computer assisted production methods (CAD/CAM) which were originally developed for the aircraft and automotive industry, have been developed for precise and repeatable production of dental crowns in dentistry [16].

In 1983 Dr. Matts Andersson developed the Procera method, now known as Nobel Biocare, and used it to create high-precision dental crowns and composite-coated restorations [17].

With the development of different materials, new surface modification techniques and digitalization, the field of dental implants is developing day by day to place implants that the patient will use for a lifetime with high success rates [18].

1.2 Dental Implants and Their Usage Areas

With the growing population, investments in the field of health are also increasing. With increased investments and research, it has been possible to improve both the average human lifespan and health conditions. Today, developments in the field of biomedicine, which receives support from many branches of science, are also gaining importance. Applications such as replacing lost organs and tissues, and therefore the need for implants, have emerged. Dental implants are biocompatible materials placed in the jawbone to restore the patient's normal function, speech, health and aesthetics in cases where natural teeth are lost due to periodontal disease, failed root canal, injury or other reasons [19].

Dental implants are the solution in case of a single tooth loss or in case of multiple tooth loss, the appropriate dental implant is selected in line with the patient's needs and health status.

Dental implants also offer higher functionality in cases where conventional removable prostheses are not sufficient or a complication occurs [20].

1.3 Types of Dental Implants

In the selection of the appropriate implant for the patient; patient profile, implant material selection and implant design (shape and dimensions), quality and quantity of tissues, and implant surface modifications should be considered [21].

According to their placement, dental implants are divided into three groups: endosteal, subperiosteal and transosteal [22].

1.3.1 Subperiosteal implants

Subperiosteal implants are implants that are placed between the bone and the gingiva after measuring over the surgically opened bone suitable for the patient [23].

These implants, which distribute the chewing force throughout the bone, are mostly used in cases where the endosteal implant is not suitable for placement. Subperiosteal implants may offer advantages such as low cost and short treatment duration compared to alternative treatment methods to endosteal implants [24].

1.3.2 Transosteal implants

Transosteal or mandibular stable implants are used in deformed edentulous or partially edentulous patients who lack adequate bone support [25]. The transosteal implant system consists of a metal plate placed at the bottom of the mandible, screws passing through the jawbone, and columns embedded in the gingiva [26].

These implants, which can only be applied to the lower jawbone, are expensive as they need to be made according to the patient's profile, and these implants are rarely used. The transosteal implant image is given in Figure 1.1 [27].

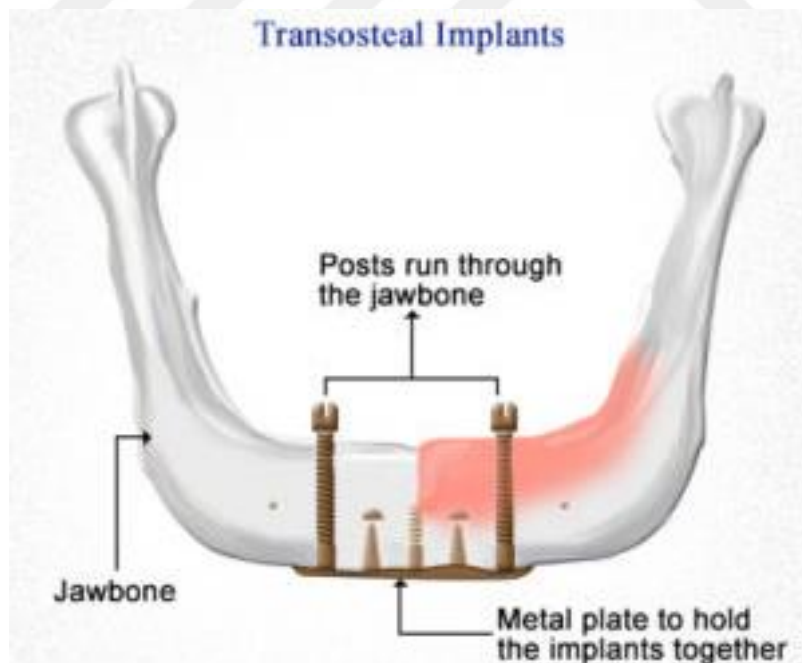


Figure 1.1 : Transosteal implants [27].

1.3.3 Endosteal (Endosseous) implants

Endosteal type implant first successfully placed by Branemark in 1965. Endosteal implants, which were initially placed after waiting for healing after tooth extraction, were accepted after Lazzarra's successful implant placement immediately after tooth extraction, confirming their reliability with many studies in the following years [28].

Endosteal or endosseous implants are single implant units that are placed in the jawbone and imitate the lost tooth root [29]. There are 3 types of endosseous implants: blade, cylinder and screw type. Screw-shaped endosteal implants are the most commonly used implants today. Endosteal implant types are shown in Figure 1.2 [30].

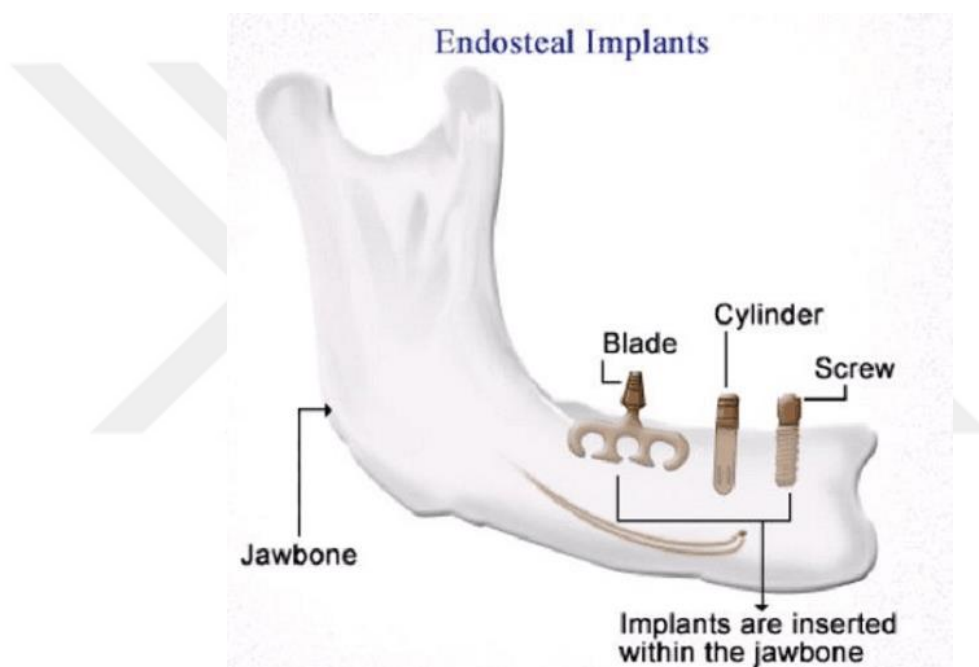


Figure 1.2 : Endosteal implant types [30].

1.4 Materials Used in Endosteal Implants

The two most important features that a biomaterial to be used in the biomedical field should have is biocompatibility and biofunctionality. Biofunctionality can be defined as the medical device or material inserted as the expected function, and the biocompatibility can be defined as the compatibility of the material with the body [31].

The ultimate goal after endosteal implant placement is to provide osseointegration or biointegration. Many materials and surface modifications are used for this purpose, but only a few of the materials used provide osseointegration or biointegration [32].

Various materials used in endosteal implant materials are given in Table 1.1 [33].

Table 1.1 : Various materials used in endosteal implants [33].

Implant Material	Common Name & Abbreviation
Titanium	cpTi
Titanium Grade 5	Ti6Al4V
Stainless Steel	SS, 316 L SS
Cobalt Chromium Alloy	Vitallium, Co-Cr-Mo
Gold Alloys	
Tantalum	Ta
Alumina	Al ₂ O ₃
Hydroxyapatite	HA
Beta-Tricalcium Phosphate	Ca ₃ (PO ₄) ₂ , β -TCP
Carbon	C
Carbon-Silicon	C-Si
Bioglass	SiO ₂ , / CaO / Na ₂ O / P ₂ O ₅
Polymethylmethacrylate	PMMA
Polytetrafluoroethylene	PTFE
Polyethylene	PE
Polysulfone	PSF
Polyurethane	PU

Dental implant materials include materials that have different interactions with the body. In the first stage after implant placement, primary stability occurs between the bone and implant interface due to this interaction. In the weeks following the surgical placement of the implant, bone remodeling takes place around the implant and finally new bone formation occurs [34].

1.4.1 Metals

Metals are thought to be the oldest used material among dental implants and are still the most commonly used dental implant material today. Metallic biomaterials used in the dental implant field include pure titanium and titanium alloys, cobalt-chromium alloy, gold alloys and stainless steel [35].

Gold, silver and platinum and their alloys are frequently used in dentistry because of their good castability, ductility and corrosion resistance properties [36].

However, while corrosion resistance is important among metallic biomaterials, it is also important that the effect the corrosion has on the tissue and the metallic products released from the prosthesis due to wear do not damage the tissues [37]. Therefore one of the most important considerations for the selection of suitable materials for metals

is biocompatibility. Since biocompatibility is related to the corrosion of metals in the body, the use of structures such as tantalum, zircon, and platinum as implant materials is limited. When we consider chromium cobalt mixtures, despite their good mechanical properties, their high melting point and low density cause difficulties in castability. Gold alloys, which have better mechanical properties than pure gold, are materials that can be used in dentistry due to their stability and high corrosion resistance [38]. Titanium and its alloys are an excellent choice as implant material due to their high specific strength, high corrosion resistance and biocompatibility provided by low electrical conductivity [39]. Today, titanium and its alloys are the most widely used implant materials [40].

1.4.2 Polymeric materials

Today, the most common titanium and its alloys are used as implant materials due to its high biocompatibility and corrosion resistance and excellent mechanical properties. Polymeric materials such as HPPs, Methyl methacrylate (MMA), Polyethylene Glycol (PEG) and Polyether Ether Ketone (PEEK) are also used for dental and medical applications. Materials such as HPP and PEEK have the positive properties required for implants and clinical studies are ongoing for their potential applications [41].

1.4.3 Ceramics

Bioceramic materials such as alumina, calcium phosphates, hydroxyapatite have high biocompatibility, but their low ductility limits their use as implant materials. It has been reported that these materials support new bone formation and osseointegration after implant placement. Among these materials, it has been observed that titanium implants coated with Hydroxyapatite perform chemical bonding [42].

Zirconia has high chemical stability and corrosion resistance, making it suitable for dental implant material.

The use of zirconia as a dental implant material has advantages such as higher hardness and lower modulus than alumina [43]. Case reports using single-piece rough-surfaced zirconia implants have reported excellent esthetics and remarkably good results in some single and multi-rooted tooth deficiencies [44].

1.5 Requirements of Dental Implants

For titanium implants to be clinically successful, they must be corrosion resistant, biocompatible and have suitable physicochemical properties [45].

Implant materials have a large market in line with increasing diseases and needs. Implant materials are prepared by selecting materials such as bone, tissue, polymer, metal, ceramic according to the needs and wishes of the user. Implants are exposed to many factors such as bacteria, corrosion, changes in surface chemistry in the biological environment [46]. Dental implants, like many other implant materials, have important requirements to meet because they have direct contact with vital tissues and are exposed to chemical and mechanical bio-environments. Dental implant materials should be biocompatible with the body and should not cause side effects, discomfort and pain [47]. Implant material should have desirable mechanical properties to ensure uniform distribution of stress and reduce mobility at the implant-bone interface. It is important to choose implant material with a modulus of elasticity similar to bone tissue [48]. The surface roughness of the implants is another factor affecting the clinical success of dental implants, increasing the roughness and increasing the contact area at the bone interface can improve the attachment of the implant to the bone [49]. Requirements of implant materials are shown in Figure 1.3 [50].

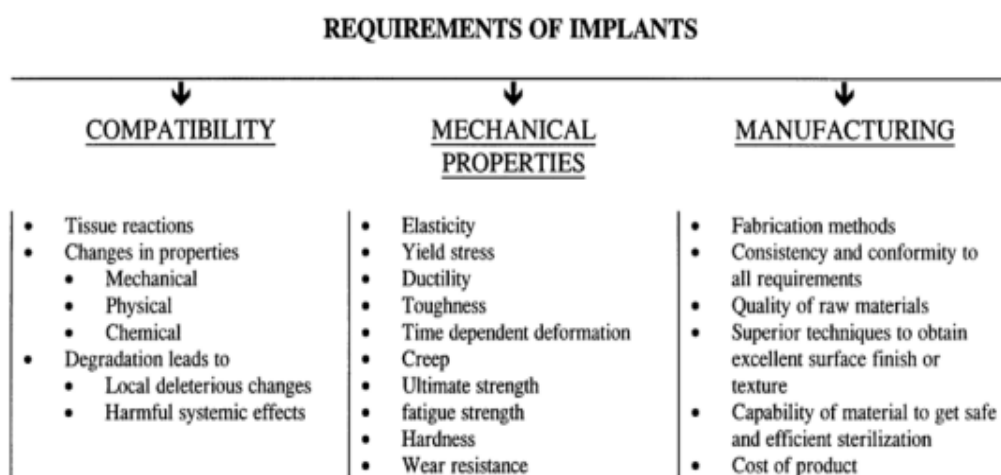


Figure 1.3 : Requirements of an implant material [50].

2. BIOCOMPATIBILITY OF DENTAL IMPLANTS

Many materials such as fillings, different shapes and types of implant materials, grafts are used in dentistry. The addition and development of new materials to these materials over time has also led to increased awareness of the biological risks of these materials. There are many definitions in the literature for the definition of biocompatibility. In general, biocompatibility can be defined as the compatibility of the material with the tissue and the ability of the applied material to exhibit a conformable host response [51]. Therefore, the need to evaluate the safety of the material before its clinical applications has arisen. The term "host response" encompasses a wide range of biological responses ranging from cytotoxicity and allergic reaction to biostimulatory effects on tissues [52].

Dental implants, which are an area where many disciplines work together, have important features to have because they are in contact with many oral tissues. Dental implants must be biologically, mechanically and morphologically compatible with the tissue. It is important that it is biologically compatible, does not cause toxic effects, is mechanically compatible, stress distribution is as equal as possible, and morphological compatibility promotes bone development in the area where the implant is placed [53].

2.1 Alveolar Bone

Alveolar bone is a tissue that is part of the maxilla and mandible, surrounds the root of the tooth, fixes it in place, and undergoes continuous bone remodeling. The alveolar bone consists of two parts, the Alveolar Bone Proper and the supporting bone. Bones are divided into 4 types according to their density [54].

2.1.1 Classification of bone density

Lekholm U and Zarb GA classified bone density into 4 groups depending on the amount of cortical and trabecular bone [55].

Type 1 bone is thick homogenous cortical bone. Type 2 is a type of bone that consists of dense trabecular bone and surrounding by cortical bone. Type 3 bone consists of

dense spongy bone surrounded by a small amount of cortical bone. Type 4 bone consists of low-density trabecular bone and a thin layer of cortical bone surrounding it.

Similarly, Misch CE defined 4 bone types according to the bone density in the edentulous parts of the mandible and maxilla [56].

D1: This type of bone is dense cortical bone

D2: Consist of high density porous cortical bone and coarse trabecular bone

D3: Trabecular bone sorrounded by porous cortical bone

D4: Low-density trabecular bone and very thin cortical bone

Later, Misch CE divided bone density into 4 categories according to the resistance of the bone during drilling. Bone density in maxilla and mandible according to Misch is given in Figure 2.1 [57].

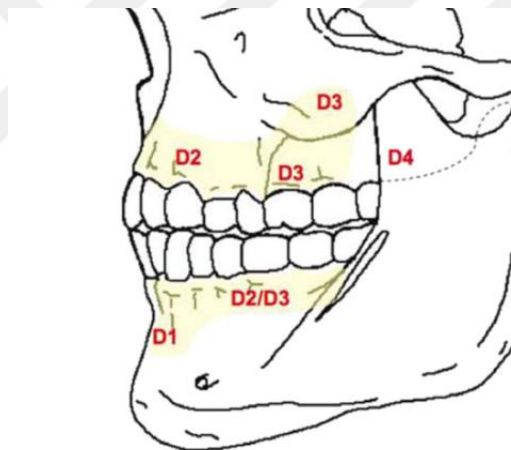


Figure 2.1 : Bone density and region according to Misch [57].

D1: Similar to drilling into maple or oak wood

D2: Similar to drilling into white pine or spruce wood

D3: Similar to drilling into balsa wood

D4: Similar to drilling into styrofoam [57].

2.2 Osseointegration

The foundations of today's modern implantology was laid by the Professor Per-Ingvar Branemark [58]. Branemark and coworkers performed in vivo microscopic studies and

discovered that the screw shaped implant was surrounded and incorporated with the living bone tissue in which it was placed. Later, he termed this phenomenon osseointegration [59].

Later, osseointegration was defined as the direct anchoring of an implant with the formation of bone tissue around the implant without fibrous tissue growth at the bone-implant interface [60]. Since osseointegration is the link between the implant and living bone tissue, it is very important for the mechanical stability of implant and successful implant planning [61].

2.3 Stages of Osseointegration

Osseointegration involves mechanisms similar to primary bone healing. Drilling and the accompanying implant placement into endosteal location initiates a process similar to wound healing [62]. The osseointegration process begins with the creation of the cavity and the placement of the implant into endosteal location. Although the process of wound healing and osseointegration are similar, there is a fundamental difference between them. While healing is achieved between tissues or bones during fracture healing or wound healing, a union occurs between bone and foreign implant material in osseointegration [63]. The wound healing process can be divided into three stages; inflammatory, proliferative and maturation phases [64].

2.3.1 Inflammatory phase

The blood coming out of the vessel after the trauma that occurs in the tissue after drilling or implant placement is placed between the fixture and the bone.

As a result of damage to endothelial cells after surgical procedure; Fibrin polymerization and blood clot are formed by homeostasis mechanisms to maintain body integrity and prevent fluid loss. The formed blood clot acts as a temporary matrix for neoangiogenesis and the bone healing process. In the clot, platelets trigger a sequence of adhesion and clustering, creating a fibrin framework that supports the movement, growth, and specialization of leukocytes and mesenchymal cells. These cells migrate from the post-capillary venules to the peri-implant area within 1 to 3 days after implantation [65].

2.3.2 Proliferative phase

The inflammatory phase is followed by the proliferative phase. This phase lasts about 10-14 days, starting on the second day. In the first 7 days, vascular growth and neovascularization occur in the implant area.

Metabolism of inflammatory cells, fibroblasts, progenitor cells and other local cells formed in the previous phase triggers the transformation of mesenchymal cells into fibroblasts, chondroblasts or osteoblasts depending on the oxygen concentration in the environment. Osteoblasts produced from mesenchymal cells are rich in cells and synthesize new bone matrix. This matrix, which is formed from fibroblasts, chondroblasts and osteoblasts transformed from extracellular mesenchymal cells, is then formed into a fibrocartilaginous callus that turns into bone callus for new bone formation. Woven, i.e. immature primitive bone, continues to form for two weeks after implant placement [66].

2.3.3 Maturation phase

The proliferative phase is followed by the maturation phase. In this phase, the primitive woven bone formed in the previous phase is replaced by mature lamellar bone. Osteoclastic resorption accomplishes this change, initiating the bone remodeling phase. After remodeling, the callus bone structure has a regular lamellar shape [67-68].

2.4 Factors That Effect Osseointegration

Osseointegration is affected by various parameters such as implants shape length and diameter, surface characteristics, bone density and implant surface area, loading forces, surgical techniques [69].

A study revealed that the success rate of dental implants is influenced by factors such as the materials, design, and surface finish of the implants; the condition of the bone; surgical procedures; and the loading conditions applied to the implants [70].

2.4.1 Effect of bone density

Bone quantity and quality can directly affect lot of factors such as implant length, diameter and type, surgical approach , healing time, loading torque and implants placement angle which are crucial for succesful implant planning. [71]

As a result, for a successful implant planning, it is important that the bone density and quality are sufficient, and the amounts of the cortical and trabecular structure of the bone are sufficient [72].

As we stated before, D1 bone is dense cortical bone and its hardness is similar to oak or maple, while D2 bone has a thin trabecular bone structure surrounded by dense cortical bone and is similar in hardness to white pine. Since D1 bone is harder than the others and contains fewer blood vessels, care should be taken against overheating during surgical implant placement. Type D2 bones have a lot of blood in contact with the implant surface [73]. Although D3 bone is weaker than D2, it is classified as a dense trabecular bone with sufficient strength, surrounded by thin cortical bone, and similar to balsa wood in terms of hardness. D4 bone has thin trabecular bone and insufficient cortical bone, has low mechanical strength and is similar to Styrofoam in hardness [74].

The bone density is important as it influences the area in contact with the implant surface and stress distribution. In the case of low bone density, the stress acting on the bone grows. It has been shown that more implant bone contact and higher implant success are obtained in Type 1, 2 and 3 bones with higher bone density [75].

Similarly according to many studies, high implant success has been achieved in d1, d2 or type 1, type 2 bone with dense cortical bone. In a study conducted by Jaffin et al. on 1045 implants, no difference was observed in the success of implants placed in type 1,2,3 bone, while 35 percent failure was observed in type 4 bone. Since there is a relationship between bone density and implant success, computed tomography is currently used to determine bone quality assessments [76].

2.4.2 Implant design

Diameter, length and design features of dental implants are important in terms of providing and maintaining both primary stability and osseointegration. One of the important parameters for ensuring stability at the bone-implant interface is to provide equal stress distribution by using these properties according to the patient profile. Among the most important factors affecting stress distribution are the diameter, length, shape and surface structure of the implant. A more homogeneous stress distribution can be achieved with an increase in the diameter or length of the implant. But the most important factor influencing stress distribution is diameter [77].

In a finite element analysis by Anitua et al. a stress value of 122.9 MPa was determined for the 2.5/8.5mm implant, while a stress value of 39.6 MPa was found for the 5/15mm implant. Increasing the diameter reduced the stress by 31%, while increasing the implant length provided a 1.7% reduction in stress [78].

According to a study by Eric et al. investigating the effects of different implant diameters, stepped and straight implants on the stress distribution with finite element analysis; It has been found that choosing a wide implant diameter suitable for the patient profile and within morphological limits when choosing the implant causes less stress on the bone surrounding the implant, and at the same time, stepped cylindrical implants are more successful than flat implants in terms of equal stress distribution [79].

Another factor affecting osseointegration is the surface area of the implant. It greatly affects the response of tissues to the implant. The increase in surface roughness creates a higher implant-bone area than smooth surfaces and promotes osseointegration. Also the roughness of the surface provides better adhesion of the implant to the bone and less stress around the implant with the increased bone surface [80].

In a study by D. Buser et al. on miniature pigs, rough surfaces showed higher bone apposition than polished or fine-structured surfaces [81]. Wong et al. showed the importance of implant surface roughness in obtaining mechanical fixation as a result of their experiments [82].

2.4.3 Loading protocols

The loading method of the implant can affect the success and osseointegration of the implant. Loading protocols for implants can be divided into two categories: conventional and immediate. Loading protocols are shown in Figure 2.2 [83].

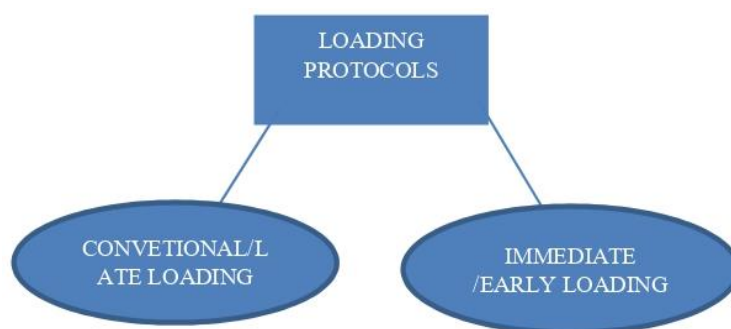


Figure 2.2 : Loading protocols [83].

In the conventional loading protocol, it is based on performing the loading after 3-6 months for the mandible and 6-8 months for the maxilla, waiting for osseointegration to occur before implant loading [83]. In immediate or early loading protocols, the implant loading process consists of a single step. In this way, it provides a more comfortable and cheaper process [84].

Dental implantology has evolved through many stages over the years. Dental implantology can be divided into three parts according to their periods and loading protocols: Pre-Branemark era, Branemark era, Post-Branemark era. In the pre-Branemark era, loading of dental implants was done at the time of insertion. In this way, it was concluded that crestal bone loss could be prevented [85-86].

Branemark et al., who defined osseointegration and laid the foundations of modern implantology, described a surgical method consisting of two parts. This protocol consisted of the opening of the bone to be implanted, the formation of tissue on the embedded and unloaded implant for 3-6 months. With this loading after healing approach, the risk of infection was reduced and micro movements that could slow down healing were also prevented [81].

In the later period, some researchers reported that osseointegration could be achieved with the use of root-shaped implants. It has been reported that osseointegration can occur even if these implants are used throughout the soft tissue during early bone remodeling. With the use of root-shaped implants, the disadvantages such as the two-stage surgery protocol required by the other method, the discomfort it causes and its long-term duration have been reduced. However, 3-6 months must be waited for the placement of the prosthesis [87].

With the advances in dental implantology, the mechanism of osseointegration has been better understood and has led to a focus on bone healing. With the development of the immediate loading protocol, it was aimed to ensure that the missing teeth perform their functions quickly and to improve the aesthetic appearance [88].

In the compilation of studies by Emeka Nkenke et al., in which many parameters were compared, no significant difference was found between conventional and early loading in terms of plaque index, bleeding index, implant stability and implant success rates [89].

In the systematic review and meta-analysis of clinical studies by Chen et al., the implant success rate was comparable between early loading and immediate loading, while the meta-analysis results reported a higher risk of failure in the test group when comparing traditional and immediate loading protocols [90].

When selecting immediate or conventional loading protocols, factors such as primary implant stability, shape and characteristics of the implant, patient bone characteristics and density, patient health status and habits, and loading forces should be considered [91].

2.4.4 Surgical procedure

Implant material should not come into contact with any material before insertion and should be placed sterile. The clinic where the surgical procedure will take place should be sterile. For the long-term success of the implant, the surgeon who chooses the implant should pick the implant material and method suitable for the patient's profile [92].

Likewise, while creating the cavity in which the implant will be placed, attention should be paid to avoid excessive temperature rise in the bone. In case of exposure to a temperature above 43 °C for one minute, bone cells irreversibly lose their vitality. These factors are important for the success of osseointegration [93].

3. TITANIUM DENTAL IMPLANTS

Dental implants are artificial teeth that are placed in place of missing teeth that have been lost for various reasons. The history of dental implants dates back more than 2000 years, but the use of modern dental implants began with dental restorations performed during World War II [94]. One of the most important developments in dentistry and implantology was Branemark's definition of osseointegration. With the contributions of developing technology, the field of implants has made many advances in both medical and aesthetic terms today [95].

The use of dental implants helps to eliminate deficiencies in chewing, digestion and speech functions and provides an aesthetic appearance. The most commonly used materials in dental implants are titanium and its alloys [96]. Titanium is a chemical element, with atomic number of 22 and atomic weight 47.90. Titanium is the 9th most abundant element on earth and the 4th most abundant element in the earth's crust [97]. Titanium was discovered by William Gregor in 1791 in England and later was named by Klaproth [98]. Although titanium was discovered in the late 18th century, extracting titanium from titanium-rich minerals was a handicap, and metallic titanium production was only demonstrated in the late 1930s [99]. With the discovery of the excellent properties of titanium and its alloys, the interest in titanium and its alloys is increasing day by day. Today titanium and titanium alloys are one of the most attractive materials for biomedical applications due to their mechanical properties and high corrosion resistance [100]. Today, the most commonly used 4 classes of pure titanium and an alloy are used for dental implants. Commercially pure titanium (cp Ti) is classified into four grades, Grade 1, Grade 2, Grade 3, and Grade 4, according to the ASTM F-67 technical standard for biomedical applications. Grade 5, a titanium alloy, is commonly used in prosthetics and offers low shear strength and low wear resistance [101].

Titanium exists in two crystal structures, hexagonal close-packed crystal and body-centered cubic crystal, depending on temperature. Pure titanium exists in a hexagonal close-packed (HCP) crystal known as the alpha phase at temperatures below 882 °C.

When titanium is heated above this temperature (transus temperature), titanium undergoes an allotropic phase transformation and exists in a body-centered cubic crystal, (BCC) also known as the beta phase [102]. Stable alpha, beta and alpha-beta phases can be obtained by adding certain amounts of some elements to pure titanium at room temperature. Aluminum (Al), nitrogen (N), carbon (C) and oxygen (O) act as alpha stabilizers, while manganese (Mn), chromium (Cr), iron (Fe) and vanadium (V) act as beta stabilizers. Structure and alloy content of titanium are shown in Figure 3.1 [103].

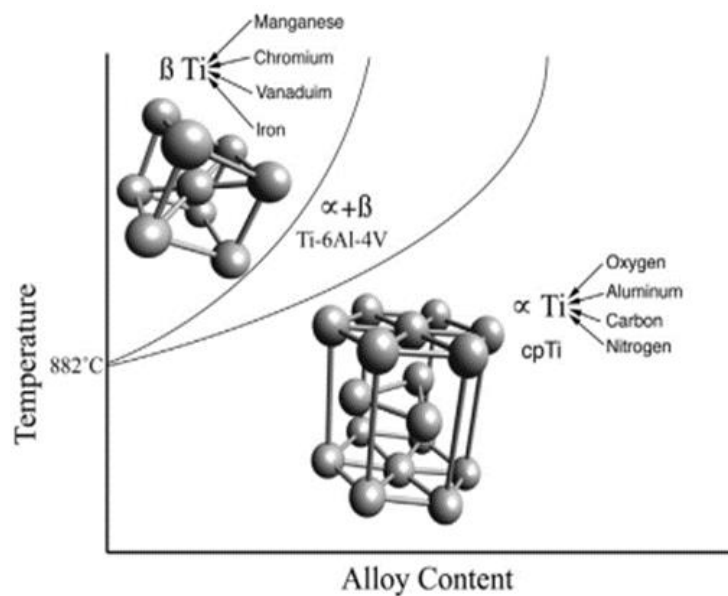


Figure 3.1 : Alpha (α) and (β) phase.

The Ti implants used in dentistry are most commonly composed of commercially pure titanium (cpTi) or titanium–6aluminum–4vanadium (Ti–6Al–4V) [29]. The cpTi is a single (alpha) phase crystal at body temperature whereas the Ti–6Al–4V is an alphabeta phase alloy. [104] Titanium and its alloys are the most preferred materials in the field of dental implants today due to their excellent mechanical properties, high corrosion resistance and biocompatibility. In addition to pure titanium, titanium alloys are often used in dental implants. Ti-6Al-4V is the most used alloy today. [105]

3.1 Properties of Titanium

Commercially pure titanium (cp Ti) has low thermal conductivity and elastic modulus, moderate strength, good corrosion resistance and high reactivity.

At low temperatures it has a hexagonal close-packed structure called alpha titanium (hcp), while at high temperatures it has a stable body-centered cubic (bcc) structure called beta titanium. Properties of cp Ti is shown in Table 3.1 [106].

Table 3.1 : Properties of titanium [106].

Property of Cp Titanium (Alpha Forms)	Value
Atomic Number	22
Atomic Weight	47.9
Density [g/cm ³]	4.5
Melting Point [°C]	1670
Thermal Conductivity [W/mK]	15-22
Hardness	100-260
Elastic Modulus [GPa]	100-145
Yield Strength (MPa)	140-655
Tensile Strength (MPa)	235-550
Corrosion Resistance Properties	High

Depend on the chemical composition and titanium content, cp titanium grades can show different valued properties. Permissible ratios of carbon, nitrogen, hydrogen, iron, and oxygen for each grade of pure titanium according to ASTM standards are shown in Figure 3.2 [107].

Table 1 - Standard ASTM number, chemical requirements (max mass%), tensile strength (TS), yield strength (YS), modulus of elasticity (E) and Brinell hardness (HB) of cp Ti, Ti alloy GS and bone properties. Based on Technical Standards ASTM F67 and ASTM F136.											
Ti grade	ASTM	O	Fe	H	C	N	Ti	TS (MPa) ^a	YS (MPa)	E (GPa)	HB
Ti grade 1	F67	0.18	0.20	0.015	0.08	0.03	Balance	240	170	100	120
Ti grade 2	F67	0.25	0.30	0.015	0.08	0.03	Balance	345	275	100	160
Ti grade 3	F67	0.35	0.30	0.015	0.08	0.05	Balance	450	380	100	200
Ti grade 4	F67	0.40	0.50	0.015	0.08	0.05	Balance	550	483	102	250
Ti grade 5 ^b	F136	0.13	0.25	0.012	0.08	0.05	Balance	860	795	115	
Cortical bone								186		5-30 ^c	

^a Mechanical properties requirements for annealed wire with diameter higher than 3.18 mm.
^b The compositional requirement Ti GS shall meet the following: Al: 5.5-6.5 and V: 3.5-4.5.
^c The bone modulus depending on the type of the bone and the direction of measurement.

Figure 3.2 : Permissible ratios of C, N, H, Fe, and O [107].

3.1.1 Mechanical properties of titanium

Titanium is renowned for its exceptional mechanical properties, making it a preferred material in various engineering applications, such as aerospace, automotive, and biomedical fields. Titanium exhibits a remarkable balance between strength and weight, providing structural integrity without adding excessive mass. This characteristic is particularly advantageous in aerospace applications where weight

reduction is crucial [108]. Commercially pure titanium has tensile strengths ranging from 275 to 590 MPa, with strength levels primarily influenced by oxygen content. Titanium alloys, such as Ti-6Al-4V, can achieve tensile strengths up to 900–1200 MPa, enhancing their suitability for high-stress applications [109]. Titanium alloys demonstrate superior fatigue resistance, enabling them to withstand cyclic loading conditions without significant degradation over time. Titanium's low thermal conductivity is beneficial in applications requiring thermal insulation or minimal heat transfer [110]. These mechanical properties, combined with biocompatibility, make titanium and its alloys indispensable in medical implants and devices, ensuring both performance and compatibility with biological tissues [111].

3.1.2 Corrosion properties of titanium

In biomedical applications, titanium and its alloys have been frequently preferred for a long time due to their excellent mechanical properties. Today, the most preferred material in the field of dental implants is titanium and its alloys. Besides the excellent mechanical properties and biocompatibility of titanium and its alloys, corrosion resistance properties are also important for the success of the implant.

Titanium can corrode at different rates depending on the conditions of the environment. For this reason, corrosion should be considered not only as a property of the material, but also as a property of the environment in which the material is present [112].

It is known that corrosion resistance has a significant effect on implant stability and biocompatibility. There are two types of corrosion: chemical corrosion (dry corrosion) and electrochemical corrosion (wet corrosion). In chemical corrosion, the metal forms a compound by directly exchanging electrons with another element in the environment through reactions such as oxidation, sulphurization, halogenation.

In electrochemical corrosion, the exchange of electrical charges with liquid electrolytes occurs. As a result of this type of corrosion, weakening of the materials and separation on the surface occur, so it is important that dental materials have high corrosion resistance [113]. Features such as variable temperature and pH, and constant humidity contribute to the formation of corrosion in the oral environment.

It is important that the material to be used has sufficient corrosion resistance against this corrosion that occurs in the oral environment. In cases where the corrosion

resistance is not sufficient, the properties of the material are adversely affected. At the same time, corrosion products can adversely affect the biological system. Corrosion resistance plays an essential role in the biocompatibility of a material [114].

Titanium and its alloys have long been preferred in biomedical applications due to their good corrosion resistance properties in the human body. Titanium is a reactive metal and has a high affinity for oxygen, a chemically resistant passive oxide film layer up to 10 nm thick is formed on the surface in contact with air. The passive oxide formed on titanium is usually TiO_2 [115]. The formation of a thermodynamically stable, fast-forming, self-repairing, protective oxide film layer on the titanium surface makes it corrosion-resistant [116].

3.1.3 Biocompatibility of titanium

Titanium and titanium alloys are frequently preferred as orthopedic and dental implant materials. In addition to its excellent mechanical properties and low density, its biocompatibility is also effective in the fact that titanium and its alloys are frequently preferred today. Biocompatibility, defined as the material placed in the body being compatible with the body and not having any adverse effects, is one of the most important features that an implant material should have. Metal ions emerging due to corrosion can cause adverse effects on tissues. Therefore, one of the most important factors affecting the biocompatibility for metal implants is the corrosion resistance of the material. As we mentioned before, the passive oxide film formed on the titanium surface after contact with air or moisture prevents corrosion. In addition to preventing corrosion, the oxide layer also plays a role in the success of osseointegration. Thick and rough oxide layers shorten the osseointegration time [117].

It has been shown that the two most widely used titanium types, pure titanium and Ti-6Al-4V alloy, can easily provide osseointegration. Both titanium types are bioactive and can support bone formation in case of direct contact with the metal surface [118]. The direct bonding of titanium and its alloys with bone without forming a soft tissue capsule is an evidence of its high biocompatibility [119].

3.1.4 Factors affecting the success of titanium implants

In order for the implant to be evaluated as successful, it is important that the implant material does not cause any discomfort or effects such as pain, infection, osteoporosis

and does not harm the patient. The main reasons why titanium is preferred for dental implants are its ability to integrate with bone (osseointegration), high resistance to corrosion and biological compatibility. These factors are also crucial to the success of titanium implants [120].

3.2 Surface Modification Techniques of Titanium Implants

Osseointegration is the direct structural and functional connection between bone tissue and the surface of implant material. And it is known that osseointegration is crucial for the stability and success of dental implants [121]. One of the most important parameters affecting osseointegration and thus the success of dental implants is the surface properties of dental implants.

Osseointegration can be increased and accelerated by changes in the surface properties, thus shortening the treatment time. With the surface modification of titanium implants, the success of the implant and the rate of bone healing can be increased even in patients with low bone quality, or bone growth can be supported after implant placement in areas with low bone density. The main purpose of surface modifications is to increase osseointegration by making changes in the surface morphology and chemistry of the implant material [122]. It has been reported that implants with rough surfaces show higher bone fixation and bone-implant contact [123].

Surface modification methods of titanium implants can be divided into 3 categories as mechanical, chemical and physical [124].

3.2.1 Mechanical methods

Mechanical surface modifications are based on the principle of obtaining surface roughness as a result of shaping the material to be used as implant material by applying physical forces on it. The most commonly used mechanical surface modifications include machining, cutting, grit-blasting, polishing. In mechanical methods, surface roughness is obtained by removing material from the surface by cutting or abrasion, or by mechanically grinding the surface using particles [125].

3.2.1.1 Machined surfaces

Machined implants are implants with slight grooves on their surfaces. These implants have an average surface roughness of 0.96 μm . Their slight surface roughness supports

the attachment and deposition of osteogenic cells in the bone matrix. Different modification methods have been developed in order to increase the bone implant surface area and fixation by obtaining higher surface roughness [126].

3.2.1.2 Blasting

It is known that osseointegration is affected by surface roughness. The roughness created increases the stability of the implant and the attachment of the implant to the bone [127]. In grit blasting process, surface roughness can be achieved by blasting implant material with air-propelled hard ceramic or metallic particles [128]. The material in the sandblasting process should be selected from materials that do not prevent osseointegration and are biocompatible with the body. Particles such as aluminum oxide, titanium dioxide, hydroxyapatite, calcium phosphate, etc. are often used in grit blasting [129]. Different roughness values can be obtained by adjusting the particle size, blasting time, distance, pressure and angle [130]. Surfaces roughened with grit-blasting provide a higher success rate than machined surfaces.

The authors demonstrated the usability of biphasic calcium phosphate particles to roughen the titanium surface and found that this offered a non-cytotoxic perspective to mouse osteoblasts [131].

3.2.1.3 Polishing

In the polishing method, an abrasive material is used to contact the implant surface. When using the polishing method, a lubricant is used. The first process is carried out using abrasive paper, and then using a finer abrasive material. The polishing method is usually carried out using materials such as silicon carbide, alumina or diamond to obtain a smooth surface with Ra values of about 0.1 μm [132].

3.2.1.4 Cutting

Cutting is the roughening of the metal surface with a carbon separator. The degree of roughness can be controlled by adjusting the cutting speed and pressure. It has been reported to cause mechanical deformation in titanium and titanium alloys, so it is not a commonly used method today [133].

3.2.2 Physical methods

Physical surface modification methods are methods that do not use chemical reactions. Physical methods are listed in table 3.2 [134].

Table 3.2 : Physical surface modification methods [134].

Physical Methods	Modified Layer	Objective
A) Thermal spray; Plasma spray Flame Spray High Velocity Oxy-Fuel (HVOF) Detonation-gun Spray (DGUN)	30 μm to 200 μm of coatings (titanium, hydroxyapatite (HA), aluminum oxide (Al_2O_3), titanium dioxide (TiO_2) etc)	Improvement of wear and corrosion resistance and biocompatibility
B) Physical Vapour Deposition Evaporation Ion plating Sputtering	1 μm of TiN, TiC, TiCN, diamond and diamond-like carbon thin films	Improvement of corrosion properties, wear resistance and blood compatibility
C) Ion Implantation and Deposition	10 nm of surface modified layer or μm of thin film	Improvement of wear and corrosion resistance and biocompatibility
D) Glow Discharge Plasma Treatment	1 to 100 nm of surface modified layer	Cleaning, sterilizing, oxidizing the surface Removal of the native oxide layer

3.2.2.1 Thermal spray method

In the thermal spray method, the material to be coated is converted into liquid droplets at high temperatures and then coated on the desired material. There are different thermal spray methods such as plasma spray, flame plasma spray, high velocity oxy-fuel and detonation-gun spray [134].

3.2.2.2 Titanium plasma spray method

Titanium plasma spray method is a physical surface modification technique used to increase the surface area of the implant. Plasma spray method is based on the principle of creating a coating and roughening the surface by spraying the particles heated and accelerated by the plasma flame onto the titanium surface to be roughened [135]. The titanium plasma spray method provides an average surface roughness of 7 μm . It is known that the surface roughness increases with the plasma spray method, but it slows

down bone formation in case of formation of an oxide layer that is too thick for cell adhesion [136].

In a study on minipigs, the bone-implant interface was formed more rapidly in implants roughened with titanium plasma spray than in smooth surfaced implants [137].

3.2.2.3 Ion implantation method

The method of ion implantation is based on the principle that ions of the material accelerated in an electric field are implanted by impinging and embedding in the surface. Ion implantation is frequently used in the modification of implant surfaces [138].

D.Krupa et al. performed ion implantation surface modification with the implantation of calcium, phosphorus ions and sodium ions on titanium material. In their results obtained with the Stern method and anodic polarization curves, they reported that Na, Ca, P and Ca+P ion implantation increased the corrosion resistance of titanium [139].

3.2.3 Chemical methods

Chemical surface modification methods are based on changing the structure of the implant material surface by chemical adsorption or reaction. Methods such as anodization, acid etching, alkaline heat treatment, anodic oxidation, sol-gel, chemical vapor deposition are examples of chemical surface modifications. Today, these methods are rarely used solely, they are generally used in combination with other modification methods [140].

3.2.3.1 Anodizing method

The anodization process is based on the principle of roughening the implant to obtain micro or nanoscale oxides on the implant by exposing it to electric current. In the anodization method, changes are made in the microstructure or crystallinity of the oxide layer. In the process, the implant is in the anode position. It is possible to create different sizes of surface roughness by making changes on the anodization conditions (voltage, temperature, time, etc.) [141].

3.2.3.2 Alkaline treatment

Alkaline treatments are another method used in surface modification and have been started to be used with the aim of improving bioactivity. With the surface modification

performed using this method, bone-like apatite formation was observed on titanium implant surfaces. Alkaline treatment has several steps. First the material to be used is immersed in NaOH or KOH solution for approximately 1 day, and then rinsed with distilled water. The material, whose ultrasonic cleaning step is carried out, is then dried at about 40°C, and in the final stage, it is heated up to 800°C for 1 hour. A porous structure is formed on the surface of the titanium material, which undergoes various processes [142].

3.2.3.3 Acid etching

The purpose of acid etching is to roughen the surface by using various acids. In this method, hydrochloric, hydrofluoric, sulfuric and nitric acids are commonly used. With the acid etching process, a homogeneous rough surface and improved bioadhesion is obtained and the surface is purified from contaminants [143]. It has been shown that acid etched surfaces show higher resistance to the removal torque test than smooth surfaces, increasing the bone-implant interface [144].

3.2.3.4 Hydrogen peroxide treatment

Sodium titanate gel is formed as a result of the reaction of titanium and its alloys with sodium hydroxide. It has been reported that the formed gel layer induces the formation of apatite and provides high corrosion resistance [145].

It has been found that a titanate gel layer is formed as a result of the treatment of titanium with H₂O₂. It is thought that titanium gel coatings can improve the bioactivity of titanium implants. The H₂O₂-titanium interaction provides a process for chemical dissolution and oxidation of the titanium surface and also for the pretreatment for apatite precipitation [146].

In a study conducted by a research group, Ti-6Al-4V alloy was treated with various concentrations of H₂O₂. They observed that as a result of the modification with 15% and 25% H₂O₂ by weight, an anatase, porous titania layer was formed on the surface. They found that as a result of 25% H₂O₂ treatment, the titanium alloy had lower current density and better corrosion resistance compared with other concentrations and the untreated alloy [147].

3.2.3.5 Sol-Gel method

Sol-gel method is another chemical surface modification method, and it is a technique that enables the formation of thin layers on the implant by converting a liquid with a certain composition into a solid-gel phase.

Titanium and hydroxyapatite coatings can be produced with this method. The method is low cost and can be combined with dip coating, spray coating and spin coating [148].

3.2.3.6 Chemical vapor deposition

Chemical vapor deposition is a method based on the logic of forming a film on the material as a result of the chemical reaction of the gas phase components with the volatile precursor. Chemical vapor deposition, a method in which evaporated materials are condensed and deposited, is widely used to produce thin films. Generally this method is used in titanium and its alloys to improve mechanical and biological properties [149].

There are different varieties of chemical vapour deposition methods which are atmospheric-pressure chemical vapor deposition (APCVD), low pressure vapor deposition (LPCVD), laser enhanced chemical vapor deposition (LECVD), plasma enhanced chemical vapor deposition (PECVD), plasma-assisted chemical vapor deposition (PACVD) [150].

In a study conducted by Movassagh Alanagh et al., it was reported that TiSiN nanocomposite coating applied on Ti-6Al-4V alloy using the pulsed-DC plasma-assisted chemical vapor deposition (PACVD) method showed high corrosion resistance and wear resistance [151].

3.2.3.7 Laser ablation

Laser ablation technique is performed to improve the integration of titanium implants into the surrounding soft tissue. In the laser microprocessing step, a model of micro-nano-scale channels is created in the neck of the dental implant. In this way, the attachment of connective tissue and bone is supported and epithelial downgrowth is inhibited [152].

3.2.3.8 Sandblasting & acid etching

The SLA process is a method in which sandblasting and acid etching are carried out together. After the macro-rough surfaces created by the sandblasting process, pits of different sizes are created on the implant material as a result of the acid etching process, depending on the etching conditions [153].

A study in an animal model showed that acid etching after sandblasting increased biomechanical properties and removal torque values compared to a machined and acid etched surface [154].

Despite its success in roughening the surface and obtaining good results SLA surface modification has disadvantages in getting rid of surface contaminants. Particles such as aluminum oxide may become embedded in the surface during the sandblasting process and may not be sufficient to remove these particles as they do not dissolve in acid [155]. Implants with SLA endosseous surfaces achieve greater bone contact than the rougher TPS (titanium plasma sprayed) surfaces [156].

3.2.4 Coating applications

It has been reported that the biocompatibility, osseointegration and antibacterial/therapeutic effect of the coatings on the implant are important factors for the long-term success of dental implants. New coating applications are divided into two categories as organic and inorganic coatings, depending on the nature of the applied material.

Organic coatings include growth factors-based coatings and methods of extracellular matrix (ECM) deposition. Growth factors are molecules involved in cell growth and tissue proliferation, each of which recognizes a specific membrane receptor. Their union initiates or inhibits cell division. Bone morphogenetic proteins (BMP) and vascular endothelial growth factor (VEGF) are generally used in implant coatings. Hydroxyapatite, calcium phosphate, magnesium, graphene, carbon nanotube, nanodiamond, silver-based and bioglass coatings are among the inorganic coatings used today. Among these, nanostructured calcium, calcium phosphate and hydroxyapatite coatings are the most commonly used implant coating methods. They promote bone healing by releasing calcium and phosphate ions. It has also been reported that inorganic coatings affect the transmission and distribution of stress to the bone [157].

3.2.4.1 Calcium phosphate biomimetic coatings

Calcium phosphate ceramics have bioactive properties, so they interact with the surrounding bone and form a bone bond with the tissue. First, Groot and Geesink introduced CaP coatings for orthopedic implants and dental implants. Since then, many studies have reported that CaP coatings increase bone-implant contact, promote bone growth and increase implant fixation. Different methods such as plasma-spray, biomimetic and electrophoretic deposition are used to coat the implant surface with CaP [158].

The most successful method for coating implant surfaces with CaP is the plasma-spraying method. However, this method has disadvantages such as the need for a pretreatment due to poor adhesion properties if the coatings are thick, the formation of undesirable phases due to high temperature differences caused by the plasma, or poor control over thickness and morphology [159].

Due to the disadvantages of the plasma spray method, alternative methods have been sought. For this reason, a new coating method, biomimetic method, inspired by the natural biomineralization process has been introduced. The biomimetic method is a way to grow calcium phosphate coatings on titanium surfaces [160].

There are several methods for accelerating the precipitation of coatings from aqueous solutions. The first method involves electrodeposition of calcium phosphate using a current, a titanium cathode, and a platinum anode. This method offers advantages such as being able to control the coating thickness, short processing time and high reproducibility. Another method is based on the precipitation of calcium phosphate immersed in simulated body fluids on the titanium surface. As a result of this method performed at physiological temperature, bone-like crystals nucleate and grow heterogeneously on the implant surface [161].

3.2.4.2 Hydroxyapatite coatings

Hydroxyapatite is known to improve osteoblast proliferation and increase bone formation. Hydroxyapatite provides bone-implant fixation and promotes bone growth, so hydroxyapatite coatings have been used for a long time to increase the rate of osseointegration [162].

3.2.4.3 Extracellular matrix deposition

The extracellular matrix consists of two main extracellular proteins, fibrous and proteoglycans [163]. Components of the extracellular matrix help to regulate cellular processes and responses, influence migration, adhesion and differentiation of bone-forming cells [164]. In the proliferative step, which is among the osseointegration stages, fibroblast growth factors stimulate the fibroblast to secrete various ECM proteins. These proteins include elastin, collagen chondroitin sulfate, fibronectin, hyaluronan, and other proteoglycans [165]. Surface modifications with bone extracellular matrix components aim to increase the biocompatibility of implant materials [166].

In a study conducted by Raphael et al., they observed that elastin-like protein coatings can improve early-stage osseointegration and reduce aseptic loosening [167].

3.2.4.4 Growth factor-based coatings

One of the properties expected from an ideal implant material is that it supports both bone integration and rapid cell growth and differentiation. The main goal in modifying the implant material with growth factors is to increase the success of the implant. Bioactive coatings support bone integration and cell differentiation and growth, thus increasing the success of the implant. It is important that growth factors not only be included in bioactive coatings, but also ensure their controlled and prolonged release [168]. The main growth factors used in implant coatings to perform implant surface modification are bone morphogenetic proteins (BMPs) and vascular endothelial growth factor (VEGF). Vascular endothelial growth factor (VEGF) is a signal protein involved in both angiogenesis and vascular homeostasis. VEGF regulates angiogenesis, proliferation of osteoblasts, and bone turnover [169]. Bone morphogenetic proteins (BMPs) are one of the growth factors and they have a great importance in inducing bone and cartilage formation. Bone morphogenetic proteins, which are used more and more in dental implant coatings due to their key role, can regulate osteogenic cells and stimulate bone mesenchymal stem cells to differentiate [170].

It has been reported that anodized implants coated with recombinant bone morphogenetic proteins (rhBMP2) which is used for bone regeneration in dental

implantology, and recombinant human VEGFs, could provide alveolar bone regeneration and show improved bone-implant contact in the micro-thread [171].

3.2.5 Micro arc oxidation usage in dental implants

Micro arc oxidation (MAO) is an electrochemical surface modification process that oxidizes the surfaces of metal materials using high energy arcs. Properties such as corrosion resistance and biological properties of implants can be improved by this method [172]. MAO or commonly known as Plasma electrolytic oxidation (PEO) can be considered as an improved version of anodization that allows the production of coatings on metal surfaces. However, it differs from anodization in that the coating growth process is characterized by numerous microdischarges traveling on the metallic surface [173].

Coatings obtained by MAO can offer properties such as high mechanical properties (wear resistance and microhardness), electrical durability and good heat resistance, allowing these coatings to be used in many areas [174]. These changes in mechanical properties extend the life of the metal. The procedure involves exposing a metal sample immersed in the electrolyte solution to a positive voltage by connecting it as the positive pole (anode), which causes an oxide layer to form on the surface and, as a result, anodic oxidation of the metal [175]. With the applied high voltage, dielectric breakdown of the oxide layer occurs, resulting in the formation of a micro arc. Dielectric breakdown occurs, and the Ti ions and the OH ions in the electrolyte move rapidly in opposite directions, forming TiO_2 again. This process is called MAO or Plasma Electrolysis [176].

In the MAO method, the film formation mechanism occurs in two steps. In the first step, the metal melts, and in the next step, the ions of the melted metal substrate are sprayed to the film from the discharge channels due to high temperature and pressure. Through to this process, which continues in other places, an increase in film thickness occurs [177].

3.2.5.1 Oxide growth mechanism of MAO

In the first place, when a voltage higher than the breakdown potential of the oxide film is applied, dielectric failure occurs in the oxide layer. The loss of insulation resistance and dielectric stability in the low conductivity region causes many micro arcs to form

in the oxide layer. With the deterioration of dielectric stability, electron avalanche transition occurs, which causes an increase in temperature in this region due to electron avalanche. This temperature, which reaches up to thousands of Kelvin in the central part, is surrounded by a region with a lower temperature [178].

The strong electric field causes anionic compounds in the electrolyte to be drawn into the channels. At this time, the metal in the plasma and melted from the substrate goes to the channels and is oxidized. The plasma chemical reactions in the channels cause the pressure inside the channels to increase. With the presence of the electric field, the oppositely charged ions are separated, and the cations are thrown towards the electrolyte by electrostatic forces. Then, the oxidized metal is thrown from the channels to the coating surface in contact with the electrolyte. Thus, the coating thickness increases. Finally, the discharge channels are cooled and the reaction products are deposited on their walls. This process, which repeats in separate places on the coating surface, results in an increase in the coating thickness [179]. Different coating properties and morphologies can be obtained by changing the properties such as electrolyte composition, material used, magnitude of applied current and voltage, and treatment time [180].

Long-Hao Li et al reported that MAO treatment provided improvements in physical and chemical properties and biocompatibility. They also reported that the roughness of the oxide layer and the concentrations of Ca and P ions in the oxide layer increased with increasing voltage. They also observed a significant increase in the removal torque of the treated samples compared to pure titanium samples [181].

3.2.5.2 Process parameters

MAO or PEO is a process performed at high voltages using a specific electrolyte for valve metals. A hard porous oxide layer is formed on the metallic substrate, and the elements contained in the selected electrolyte are also incorporated into the oxide layer. Since the oxide layer grows in the lower direction, it has a high bonding strength [182].

It has been shown that the physical and chemical properties of the coating formed on the substrate with MAO can be controlled by changing parameters such as the composition and temperature of the electrolyte used, the magnitude of the applied voltage and current [183].

4. RADIOLOGY IN DENTAL IMPLANTS

In 1895, German physicist Wilhelm Conrad Röntgen revolutionized the fields of medicine and physics by discovering X-rays. While Röntgen was using a Crookes tube in his experiments, he noticed that a fluorescent-painted screen placed next to the tube glowed with an invisible ray coming out of the tube. When he repeated the experiment later, he realized that the screen responded to the production of an invisibly transmitted form of energy. He named this new ray, whose nature was unknown, "X", which is used for unknowns in mathematics [184]. With the discovery of X-rays, the first imaging method, radiography, also led to the emergence of the science of radiology. The penetration feature of X-rays is used in imaging; X-rays passing through the tissue create an image over an area [185]. X-rays reflect at different rates as they pass through tissues with different atomic numbers, thicknesses and densities in the body, creating images in different tones from black to white on the X-ray film. The higher the atomic number, thickness and density of the tissue, the higher the absorption. The parts of the film that are most affected by X-rays appear black, while the parts that are less affected appear white. Air appears black, fat tissues appear greyish close to black, water and soft tissues appear greyish, bones and calcifications appear greyish near white, and contrast material and metals appear white [186]. Radiography examinations are frequently preferred today due to reasons such as containing a lot of data in a single image, high spatial resolution, short shooting time and cheapness [185]. After the discovery of X-rays, image acquisition and image storage have gained importance. The foundations of digital imaging were laid with the development of computers in the 40s. Thus, analog images were replaced by digital images that could be stored and edited in a digital environment. Thanks to digital images, the ease of image storage and detailed examination of the image were provided, while the radiation rate and working time to which the patient was exposed were reduced [187]. Radiography examinations are frequently preferred today due to reasons such as containing a lot of data in a single image, high spatial resolution, short shooting time and cheapness [188].

4.1 Radiology in Dentistry

Dental implants are the most commonly used method in the treatment of patients with edentulous or missing teeth. The success of the procedure during implant placement depends on the good preparation and evaluation of the patient. Preoperative evaluation is a multi-stage process and is based on the evaluation of the holistic health and dental history and dental treatment status [189]. Radiology is used in the dental implant field in three stages: Pre-operative, intraoperative, and post-operative phase [190]. The use of radiology in the implant field is important in order to increase the success of implant planning and treatment. In terms of implant planning, it is important to be able to determine the bone structure and density, and to evaluate the surrounding sinuses, nerves, and root structures for the success of implant planning. Correct placement can be achieved in the light of this information during the placement phase after planning. With the use of radiology, Alveolar bone density and defects can be measured. Performing the necessary evaluations for periodontal disease diagnosis can provide the opportunity to estimate the status or degree of disease progression and to predict and evaluate treatment outcomes. Although radiography cannot show cellular activities in detail, it is useful in diagnosis and treatment and its planning [191]. With the increasing use of dental implants, the incidence of peri-implant diseases, called peri-implant mucositis or peri-implantitis, which is an inflammatory condition that occurs in the soft tissues around the implants, is also increasing. If left untreated, peri-implant mucositis develops bone loss around the osseointegrated implant, resulting in peri-implantitis [192]. Peri-implant mucositis covers the cases where inflammatory reactions are only in the mucosa and is based on various clinical parameters for diagnosis. In the future, the diagnosis that results in peri-implantitis formation and bone loss is based on radiological evaluation. Peri-implantitis is diagnosed by examining the supporting bone levels in radiological evaluation [193]. Radiology plays a crucial role in both diagnosis and treatment planning. Imaging techniques provide detailed information about internal organs and tissues, helping doctors make accurate decisions. Additionally, radiological methods are used to guide certain treatment procedures. This field is an indispensable part of modern medicine.

Today, the main radiological imaging methods commonly used include:

- Intra-oral (periapical, occlusal, bitewing) and extra-oral (panoramic, oblique lateral, lateral skull) radiographs.
- Computer Tomography (CT) & Cone Beam Computed Tomography (CBCT)
- Magnetic Resonance Imaging (MRI) [194].

The most common examination is performed with bitewing in intraoral radiography. This universal technique is low-cost and practical. The images obtained in this way are 2-D, and CT scans such as CBCT are used in cases where dental diagnosis requires 3-D information [195].

4.1.1 Panoramic radiography

Panoramic radiography is one of the imaging methods used in dentistry and is the most common extraoral radiography technique which allows the examination of teeth, jaw bones, jaw joints, maxillofacial structures in one image. It is useful in evaluating changes in bone density and structure, identifying patients at risk of osteoporosis, and performing clinical evaluations such as diagnosing early symptoms [196]. Panoramic radiography is narrow-beam rotational tomography that uses two or more centers of rotation with a predefined focal trough to produce a radiological image of the lower and upper jaw. Panoramic radiography devices use X-rays. Similar to other classic X-ray machines, these rays pass through the tissue to be imaged and reach the detector opposite it. X-rays create a contrasting structure as they pass through tissues of different densities such as teeth, bones and soft tissues. While dense tissues such as teeth and bones appear white, soft tissues appear darker and create a contrasting structure. In this device, the X-ray tube and detector move opposite each other. While the X-ray tube moves in a semicircle around the patient, the detector scans the area around the teeth, thus creating a panoramic image of the entire jaw structure in a single frame. Tooth decay is a leading cause of tooth loss. Therefore, it is important to detect caries lesions as early as possible for the management of dental caries. Periodontal diseases have been reported to be the second reason for tooth extraction. Panoramic radiography can contribute to the improvement of oral health by allowing the visualization of Dental caries, Periodontal diseases, Periapical diseases and even tumors and cysts [197].

Panoramic radiography has the advantages of low cost, low radiation dose, and obtaining a comprehensive overview in a single image of anatomical structures such

as dental arches, jaw bones, as well as alveolar nerves and maxillary sinuses that need to be protected during an operation [198]. However, panoramic radiography alone may be inadequate for diagnosing periapical lesions, bone loss, and caries, so full-mouth scanning is possible when combined with intraoral radiography. Intraoral radiography is preferred when limited areas need to be examined [199]. And it is also difficult to obtain good radiological images of some patients who have anatomical problems such as skeletal diseases, facial asymmetry or inability to maintain spinal position, and who cannot remain still during exposure due to pathological conditions such as tics, tremors or seizures [200].

4.1.2 Intraoral radiography

Every radiographic examination should be evaluated on a patient-by-patient basis, and the potential benefit should be weighed against the radiation risk. The principle of justification is the principle that emphasizes that every situation in which radiation is used must be necessary and that the benefits to the patient must outweigh the potential risks. Thus, it is aimed to prevent unnecessary radiation use [201]. The ALARA (as low as reasonably achievable) optimization principle means that the radiation load should be kept as low as possible [202]. Later, ALADA (as low as diagnostically achievable) and ALADAIP (as low as diagnostically achievable and individualized to the patient) were introduced. These abbreviations basically mean that each exposure should be justified and adapted to the patient. The basic aim of all limitation principles is to protect the person from radiation by using as little radiation as possible [203].

Intraoral radiography is another imaging method, taken by placing image receptors in the oral cavity. This technique, which provides detailed images of the teeth and surrounding tissues, basically places the film and digital sensor in the mouth. Positioning is important in this technique because the active area of the receptor is small. Various intraoral radiographs are periapical, bitewing and occlusal [204].

4.1.2.1 Periapical radiography

Periapical radiography is a widely used intraoral radiography technique and is named periapical radiography because it allows the display of two teeth and periapical tissues together. It is essential to obtain the full length of the tooth and at least 2 mm of the periapical bone on a properly taken periapical radiograph [205]. This method has many advantages such as the rapid and easy film placement, easy intraoral positioning, the

length of the image being the same and detailed as the tooth when taken correctly, and therefore sufficient for diagnosis. In terms of disadvantages; it is not reproducible, it shows bone levels poorly, it is difficult and requires an experienced operator since the correct angle must be adjusted for each patient, and proximal caries cannot be detected [206].

4.1.2.2 Bitewing radiography

Dental caries is an important chronic disease that affects the quality of life. Bitewing radiography is used to detect proximal caries that require early treatment and cannot be detected clinically by providing accurate diagnosis [207].

Bitewing radiographs can detect caries on proximal surfaces and moderate lesions on occlusal surfaces. Since Bitewing radiography has a sensitivity of 0.4 and high specificity, it can follow proximal lesions. It also allows the tracking of lesions when individual holders are used [208].

4.1.2.3 Occlusal radiography

Occlusal Radiography is one of the types of intraoral radiography used in dentistry and is generally used to obtain a wide view of the jaw, teeth, and surrounding tissues.

This technique uses a photostimulable phosphor plate to obtain occlusal radiographs. Different plates are used depending on the anatomy to be imaged [209].

Occlusal x-rays demonstrate the floor or roof of the mouth. The resulting image can show teeth that have not yet emerged, congenital cracks or fissures in the jaw, development of teeth, and abscesses [210]. Occlusal radiography is useful in areas such as visualizing roots and impacted teeth, determining the presence of cysts and odontomas, obtaining images of patients who have difficulty opening their mouths, such as patients with trismus, and determining the location of salivary stones [211].

4.1.3 Cone beam computer tomography

Scientist Godfrey Hounsfield's introduction of computerized transverse axial scanning in 1972 led to the introduction of Computerized Tomography (CT), one of the most important inventions of modern medicine. Numerous two-dimensional images obtained in computerized tomography are processed by the computer to obtain three-dimensional images [212].

Cone Beam Computed Tomography (CBCT), also known as digital volumetric tomography (DVT), was first developed for use in dentistry in the 1990s. It is widely used in all fields of dentistry such as orthodontics, endodontics, pathology, implantology and periodontics [213]. As in other imaging methods, there is an X-ray source and a detector. In this method, the cone-shaped X-ray beam that gives the method its name is used. The X-ray source and detector on a fixed platform rotate 180–360 degrees, thus obtaining a multiplanar image [214]. It differs from classical computerized tomography in two ways. The first is the difference in imaging caused by the detector and the different features that provide data, and the other is that CT has a high-efficiency rotating anode generator, while CBCT has a low-energy anode tube [215-216]. In CBCT, image creation occurs in two stages, Acquisition and Reconstruction. In Acquisition Stage, the X-ray source and detector on a fixed platform rotate 180 degrees around the patient's head one or more times to take images. In this way, 2D images are taken from different angles. In the reconstruction stage, the basic images transferred to the computer are processed, and in this section, the reconstruction of the images is performed with both visual and geometric correction. After all slices are reconstructed, they are recombined into a single volume according to the desired section [217]. This method is a technology that offers all the advantages of the CT device without the high radiation risk, taking into account the ALARA principle and the high radiation dose to which the patient is exposed by computed tomography [218].

CBTC helps many areas in pre-implant planning, such as control of jaw bone lesions, sinus, bone and dental pathologies, determination of anatomical boundaries, reliable visualization of jaw bone neurovascularization, bone morphology, volume, structure and grafting planning when necessary, and after the placement diagnosis of post-operative complications. In this way, CBCT helps in many areas such as pre-implant planning, monitoring of the implant area and complications after placement [219].

The radiological image taken with Cone Beam Computer Tomography is given in Figure 4.1 [220].

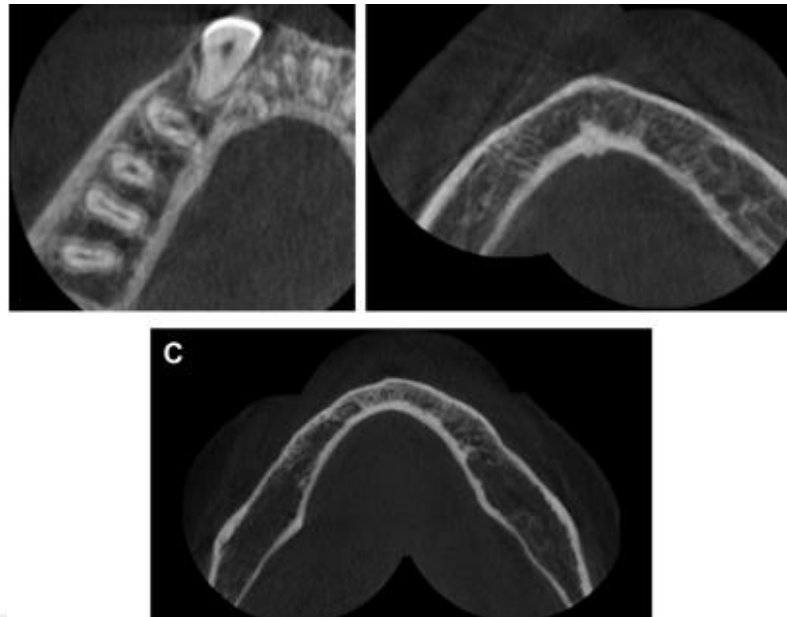


Figure 4.1 : CBCT radiological image [220].

Despite its many advantages, CBCT scans also have disadvantages such as being insufficient for wide scans due to the use of a narrow Field of View (FOV) in a limited area, making it difficult to diagnose soft tissue lesions since it shows soft tissues with low contrast, and distorting the image due to artifacts in the image caused by tissues containing metal implants [221].

4.1.4 Magnetic resonance imaging (MRI)

MRI is a non-invasive method that maps high-quality cross-sectional images in the desired plane of the body using radio frequency in the presence of a strong magnetic field. This technique does not use ionizing radiation, unlike other radiographic imaging methods. MRI uses the Hydrogen (H) atom of the water (H₂O) molecule, which is abundant in the body. Therefore, tissues with abundant water, such as the human brain, can be better visualized. It is created by measuring the signals returned from the protons excited by the magnetic field by the MRI device [222-223].

Hydrogen nucleons, consisting of a single, unpaired proton, act as a magnetic dipole. These dipoles, which are randomly oriented and have no magnetization in the absence of external influence, when exposed to an external magnetic field, as in MRI, their hydrogen nuclear axes move upward in the direction of the magnetic field and, as a result, produce magnetization. Protons spin, due to the strong magnetic field. They move in 2 ways: upward rotation parallel to the magnetic field (low energy) and downward rotation antiparallel to the magnetic field (high energy). When radio waves

are sent for a short time, they are absorbed by the hydrogen atoms. They release this energy when they return to the magnetic field. This released energy is emitted to the outside in the form of radio waves and these signals are collected by the MRI device. [224]The collected signals are processed by the MRI device in a computer environment. As a result, a detailed map of the structures in the body that are to be imaged is obtained. With the developing technology, MRI is used in dentistry in the diagnosis of temporomandibular joint diseases, inflammatory conditions of the facial skeleton, examination of the salivary glands, maxillary sinuses, masseter muscles, and detection of early bone changes such as tumors, fractures, inflammatory conditions and hematomas. [225] An image taken by a MRI device is shown in Figure 4.2 [226].

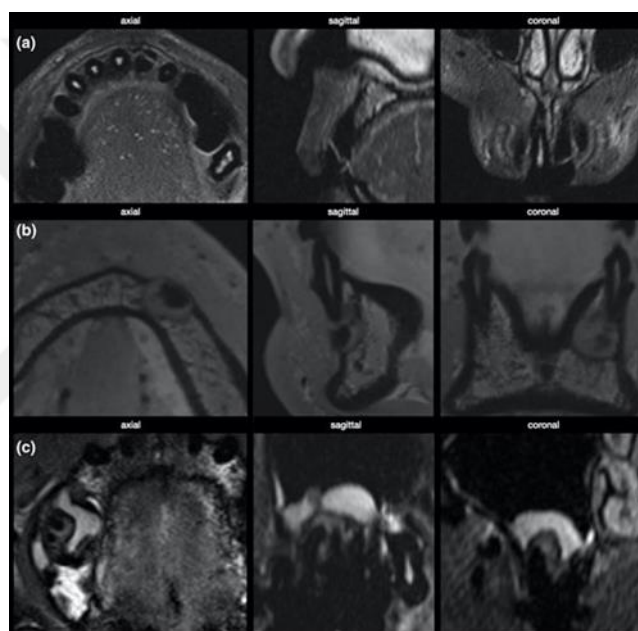


Figure 4.2 : MRI of different indications in endodontics [226].

Korn et al. studied the suitability of MRI imaging in a minipig model using MRI-compatible PEEK implants. Their findings showed that comparable results were obtained in the quantification of peri-implant bone volume and soft tissue volume, and that bone healing was analyzed in the early stage reported that it is possible. With the data they obtained, they reported that MRI could be a promising application in monitoring bone formation [227].

While MRI has many advantages, such as not using ionizing radiation and providing detailed images, it also has disadvantages such as high acquisition costs, long scanning times, changes in the magnetic field strength between dental materials and adjacent tissues, and image artifacts caused by signal loss [228].

5. EXPERIMENTAL

5.1 Materials and Methods

In this thesis study, implants made of Ti6Al4V (Grade 5) titanium alloy, which is widely used in the biomedical field and as a dental implant material, and discs cut from titanium rods made of the same metal were used. In the first stage, various implants that were previously placed in the mouth and fell out due to failure to provide osseointegration were examined. The surface properties of these implants were examined and then radiological images were taken to understand the reasons behind the fall out and failure to provide osseointegration. Afterwards, in order to investigate the effects of implant surface modifications and grit sizes on implant bioactivities and thus on implant success, sandblasting was performed using aluminum oxide grits with different grain sizes. In order to understand the difference between the implants, they were immersed in SBF before the MAO process. Then, oxide coatings were obtained by applying the MAO process. The surface properties of the samples obtained at each stage were examined. Afterwards, the implant samples and disks were immersed in Simulated Body Fluid again. After waiting for 14 days, the images were examined. The experimental studies performed are listed below.

- 1) Examination of the surfaces of the failed implant materials with SEM, then examination of their radiological images
- 2) Preparation of implant and disk materials
- 3) Surface treatments (sandblasting and acid etching) of disc and implant materials
- 4) Performing surface roughness measurements of samples
- 5) Immersion of samples in SBF and examination
- 6) Micro Arc Oxidation Process
- 7) Immersion of the samples in SBF again to observe the effects of the MAO process

8) Analysis of the immersed samples with SEM-EDS, XRD, FTIR after drying

5.2 Investigation of Failed Implants That Have Been Sandblasted & Acid Etched

In the thesis study, Ti6Al4V (Grade 5) titanium implants that were sandblasted and acid etched, placed in the mouth and failed to provide osseointegration after various periods of time were used. In the first stage, ultrasonic washing was performed to clean the surface and make it ready for SEM. SEM images of the ultrasonically washed implants were taken. Figure 5.1 shows implants that failed to achieve osseointegration.



Figure 5.1 : Implants that failed to achieve osseointegration.

5.3 Acquisition of Radiological Images of Failed Implants

Radiological images of implants were taken after SEM images were taken and surface properties were examined. At this stage, radiological images were taken to examine whether there were cracks or fractures in the internal structure of the implant samples. Radiological images were taken at the Turkish Energy, Nuclear and Mining Research Council (TENMAK). Figure 5.2 shows the radiological imaging device and imaging process.



Figure 5.2 : Radiological imaging device and imaging process.

Radiological images of implants were taken after SEM images were taken and surface properties were examined. At this stage, radiological images were taken to examine whether there were cracks or fractures in the internal structure of the implant samples. Radiological images were taken at the Turkish Energy, Nuclear and Mining Research Council (TENMAK). First, the samples to be imaged were placed on the plate and fixed. Lead numbers were placed next to the samples to prevent mixing.



Figure 5.3 : Implant samples to be radiologically imaged.



Figure 5.4 : Implant samples to be radiologically imaged-2.

A Teledyne ICM brand CP200D model X-ray generating device was used as the radiation source. Carestream brand Industrex Flex HR was used as the imaging plate, and VMI brand CR 5100 model was used as the scanner. Since the applied voltage, current and application time have significant effects on the image, these parameters were determined first. When determining the parameters, the parameter that provides the clearest image and the best visibility of the internal structure was selected. Radiological images were taken under 60 kV 7.0 mA 30 seconds. Operating parameters is shown in Figure 5.5.



Figure 5.5 : Operating parameters of the radiological imaging device.

5.4 Preparation of Disc and Implant Samples

In this thesis study, Titanium Grade 5 or Ti6AL4V was used, which is a titanium grade frequently used as implant and prosthetic material due to its high strength/weight ratio, corrosion resistance and ease of production, containing aluminum as alpha stabilizer

and vanadium as beta stabilizer. 15 identical untreated implants were used. The image of the implants used is given in the Figure 5.6.



Figure 5.6 : Untreated implants.

To prepare disk samples, disks were cut from titanium grade 5 rod. The cut disks were X mm in diameter and Y mm in length. After the cut samples were cleaned from metal oil, they were cleaned with acetone, ethanol and then ultrasonic washing. The cleaned samples were dried and made ready for sandblasting. Disc samples are shown in Figure 5.7 and Figure 5.8.



Figure 5.7 : Disc Samples.



Figure 5.8 : Disc Sample.

5.5 SLA Surface Modification of Disc and Implant Samples

For the sandblasting process, 78 nanometer (nm), 180 nm, 3-19 micrometer (μm), 63-90 μm , 90-125 μm , 150-212 μm , 212-300 μm alumina (Al_2O_3) sands and 63-90 μm silicon carbide (SiC) sands were used. Supplied sands are shown in the Figure 5.9.



Figure 5.9 : Sands of different grit sizes.

Mikrotek Dental OMK-3055 device was used to perform the roughening process using the sands provided for the sandblasting process. The sandblasting device has manual and automatic sandblasting options. The sandblasting process was carried out at 5 bar pressure. The device was cleaned before and after each sandblasting process. Sandblasting device is shown in Figure 5.10.



Figure 5.10 : Sandblasting device.

All implants that went through the sandblasting process were washed in a Protech brand ultrasonic washing machine to remove the sand grains on them. They were washed using pure water at 80 ° C for 30 minutes. The ultrasonic washing device is shown in Figure 5.11.



Figure 5.11 : Ultrasonic washing device.

The implants that went through ultrasonic washing processes were dried using a drying oven. The drying oven was set to room temperature. Drying oven is shown in Figure 5.12.



Figure 5.12 : Drying oven.

After sandblasting, the implants that have gone through the washing and drying stages are put through the acid etching stage. In this way, both the remaining sand and contaminants are removed and a clean surface is obtained, and chemical modification is carried out to create roughness on the surface. Nano-roughnesses are added to the micro-rough surface obtained as a result of sandblasting by acid etching. Citric acid and oxalic acid were used in the study. Citric acid is shown in Figure 5.13 and oxalic acid is shown in Figure 5.14.

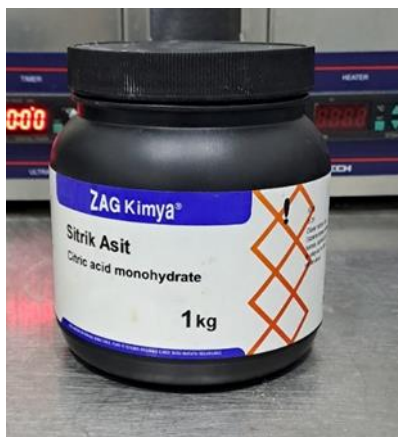


Figure 5.13 : Citric acid.



Figure 5.14 : Oxalic acid.

The apparatus used in preparing the solution is given in Figure 5.15.

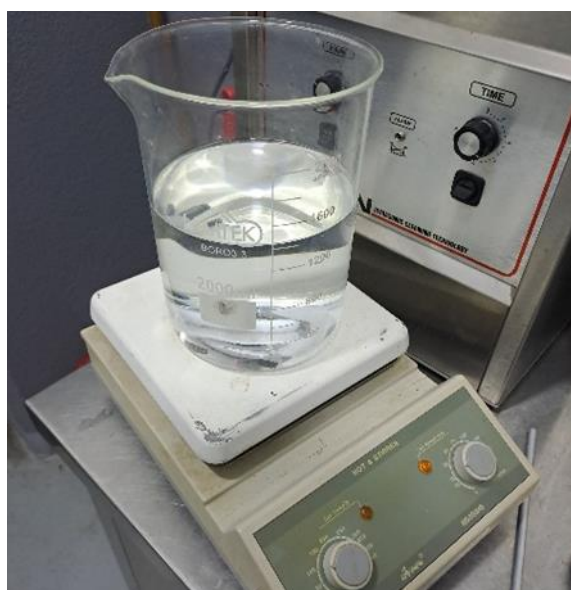


Figure 5.15 : Apparatus for preparing the solution.

Citric acid, oxalic acid and combinations of these acids are given in the Table 5.1.

Table 5.1 : Table of acids used in the experiment.

Name	Sand Particle Size	Type of Acid Used	Solution Temperature
F600 C	3-19 μm	citric acid	50 °C
F600 O	3-19 μm	oxalic acid	50 °C
F600 C+O	3-19 μm	citric acid + oxalic acid	50 °C
F180 C	63-90 μm	citric acid	50 °C
F180 O	63-90 μm	oxalic acid	50 °C
F180 C+O	63-90 μm	citric acid + oxalic acid	50 °C
F120 C	90-125 μm	citric acid	50 °C
F120 O	90-125 μm	oxalic acid	50 °C
F120 C+O	90-125 μm	citric acid + oxalic acid	50 °C
F80 C	150-212 μm	citric acid	50 °C
F80 O	150-212 μm	oxalic acid	50 °C
F80 C+O	150-212 μm	citric acid + oxalic acid	50 °C
F60 C	212-300 μm	citric acid	50 °C
F60 O	212-300 μm	oxalic acid	50 °C
F60 C+O	212-300 μm	citric acid + oxalic acid	50 °C

5.6 Surface Roughness Measurements of Samples

Measuring the surface roughness of implants is critical for biocompatibility, osseointegration, mechanical performance, infection risk, aesthetics and longevity. Appropriate roughness improves bone integration by increasing cell adhesion to the implant surface and prevents microorganism colonization. In addition, excessive roughness can lead to mechanical failures and increase the risk of wear.

Therefore, correctly optimizing the surface roughness in implant design and production directly affects the clinical success and life of the implant. At this stage, surface roughness measurements were performed after the SLA process. A profilometer was used for surface roughness measurement of implant and disk samples. The Veeco Dektak 6M brand profilometry device used is shown in Figure 5.16, and the unit where the measurements were made is shown in Figure 5.17.

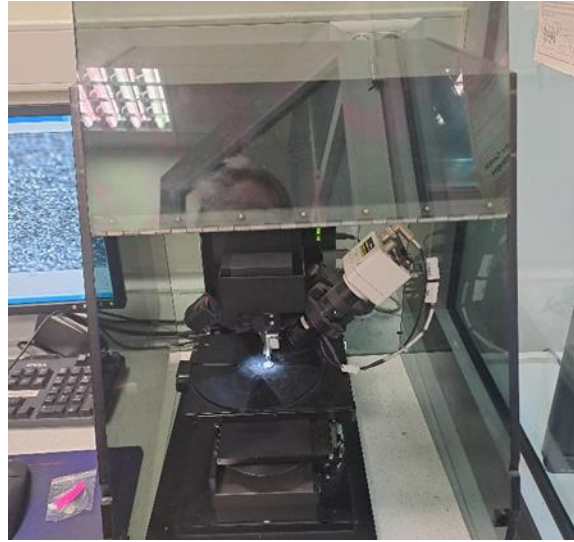


Figure 5.16 : Profilometry device.

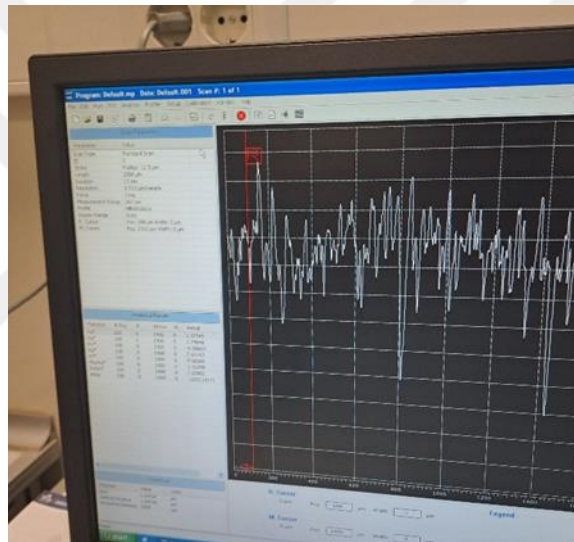


Figure 5.17 : Measurement unit.

5.7 Simulated Body Fluid (SBF)

Simulated body fluid is an artificial fluid that mimics the chemical composition of body fluids such as natural blood plasma. This fluid is used to test how the material used will react in a biological environment. SBF contains ionic compounds such as sodium, calcium, potassium, magnesium and phosphate, which are also found in blood plasma.. There is a correlation between in vivo bone growth and in vitro apatite formation ability. Therefore, in order to compare the apatite formation mechanisms disk samples and implants were immersed in SBF and examined in terms of apatite formation mechanism. The table of how many ions are present in blood plasma and SBF is given in Table 5.2.

Table 5.2 : Ion concentrations of blood plasma and SBF.

Ion	Sbf (pH 7,40)	Blood plasma (pH 7,2 to 7,4)
Na ⁺	142,0	142,0
K ⁺	5,0	5,0
Mg ⁺	1,5	1,5
Ca ²⁺	2,5	2,5
Cl ⁻	147,8	103,0
HCO ₃ ⁻	4,2	27,0
HPO ₄ ²⁻	1,0	1,0
SO ₄ ²⁻	0,5	0,5

5.7.1 Reagents for SBF

SBF provides a means to simulate how materials interact with the human body without requiring direct use of biological samples, enabling more ethical and reproducible experiments. The composition of SBF typically includes electrolytes and minerals similar to those found in human body fluids. For the preparation of SBF, materials specified in ISO 23317 were used. While supplying the materials to be used, their purity was taken into consideration.

5.7.2 Preparation of SBF

The reagent provided for the preparation of SBF, 1 liter plastic beaker, magnetic stirrer and water bath were used to prepare SBF.

The ambient temperature and liquid temperature were controlled during the SBF preparation phase. The liquid temperature was kept between $36.5 \pm 0.5^{\circ}\text{C}$ and continuous values were taken using an immersion thermometer. First, 700 ml of ion-exchanged and distilled water was added to the plastic beaker and brought to 36 degrees. Since the order of addition is important in the preparation of SBF, this order was followed and it was solved by adding from 1 to 8 in the order shown in Table 5.3.

Table 5.3 : Order of reagent materials.

Order	Reagent	Amount (depending upon purity)
1	<u>NaCl</u>	8,035 g
2	NaHCO ₃	0,355 g
3	KCl	0,225
4	(K ₂ HPO ₄ • 3H ₂ O)	0,231
5	(MgCl ₂ • 6H ₂ O)	0,311
6	c(HCl)	39 ml
7	<u>CaCl₂ • 2H₂O</u>	0,292 g
8	<u>Na₂SO₄</u>	0,072 g
9	<u>TRIS: ((HOCH₂)₃CNH₂)</u>	6,118 g
10	<u>HCl</u>	0 ml to 5 ml

A Precise ES 225SM-DR precision scale with a resolution of 0.00001 was used to perform the weighing process. Precision scale is shown in Figure 5.18. The scale was zeroed before weighing. Any sedimentation was prevented by using a magnetic stirrer and continuous stirring. After each reagent was added, it was waited until it was completely dissolved.



Figure 5.18 : Precision scale.

Ph measurement was performed before adding Tris. This value should be 2.0 ± 1.0 pH. The pH meter used is a sensitive pH meter with 0.01 resolution. pH meter is shown in Figure 5.19.



Figure 5.19 : pH meter.

5.7.3 Preparation

A liquid bath setup was prepared to be kept at a constant temperature of 37 degrees for the selected samples and discs. The discs and implants, which were placed in a tightly closed plastic container of 50 ml, were hung and immersed in the SBF and kept in the liquid bath for 14 days. Figure 5.20 shows how the implant and discs are positioned in the plastic container. During this period, the SBF was renewed every two days. Figure 5.21 shows the liquid bath.

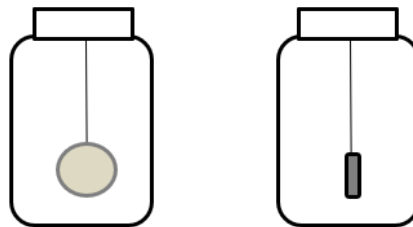


Figure 5.20 : Implant and discs in plastic container.



Figure 5.21 : Liquid bath.

After 14 days of incubation at 37 °C, the samples were removed from the SBF, cleaned with distilled water and dried at room temperature. Then, EDS-SEM images were taken.

5.8 Micro Arc Oxidation

Micro Arc Oxidation (MAO) is a process used to create hard, protective oxide coatings on the surface of metals. Under high voltage, plasma arcs are created on the metal surface to oxidize it, thus creating an oxide coating on the surface. This process is usually applied to metals such as aluminum, titanium and magnesium. MAO provides thicker, harder and more durable coatings than traditional oxidation methods. In addition, the coatings are resistant to harsh conditions such as corrosion and abrasion. In this thesis, MAO was used to improve the bioactivity of titanium. In this setup, a stainless steel container containing the electrolyte was used as the cathode and the sample was used as the anode. MAO process was carried out using 20.5 g/L Calcium Acetate ($\text{C}_4\text{H}_6\text{CaO}_4$) and 14.4 g/L Monosodium phosphate (NaH_2PO_4) in a total of 2.5 liters of solution. The process, in which 300 V positive voltage and 60 V negative voltage were applied, lasted for 3 minutes. The electrolyte container was cooled with an external cooling unit during the process to ensure that the electrolyte temperature remained below 30 °C. At the end of the process, the samples removed from the electrolyte were washed with pure water and dried.

The schematic representation of the MAO process is shown in Figure 5.22

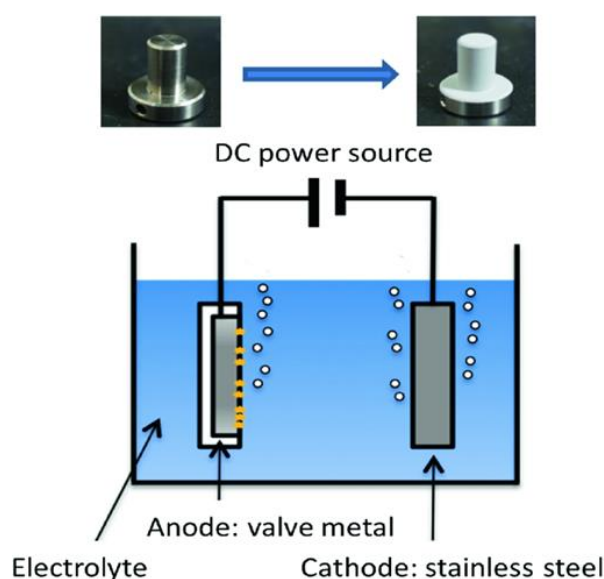


Figure 5.22 : Schematic representation of the MAO process

5.9 SEM Characterization

The Scanning Electron Microscopy (SEM) characterization of the disk and implant samples after SBF was taken at Sakarya University Faculty of Engineering. The device providing the images is JEOL brand JSM-6060I model. The microscope image is given in Figure 5.23.



Figure 5.23 : The SEM microscope image.

SEM characterization of the samples subjected to Micro Arc Oxidation was taken at ITU. The Hitachi TM-1000 model device was used.

SEM is a microscope used to obtain high-resolution images of a surface. SEM sends an electron beam to the surface of the sample and collects secondary electrons resulting from the interaction of the electrons with the sample. In this way, detailed information about the surface structure, morphology and topology of the sample can be obtained. SEM is widely used especially in fields such as materials science, biology and nanotechnology. Thanks to its high resolution and magnification capacity, it allows the examination of structures and surface features at the microscopic level. SEM is very important for understanding for the SLA surface modification has been successfully performed and for examining the surface. By using SEM characterization, micro and nano rough structures formed as a result of sandblasting and acid etching can be observed. The preparation stage before imaging is also very important.

Before each image acquisition, the samples should be cleaned appropriately using deionized water and then dried with the help of a dryer at a temperature suitable for the structure of the material. Since there is a coating on the surfaces of the sample and it will be reused for the Micro Arc Oxidation process later, no coating was applied. The images were taken by applying 15 kV.

5.10 X-ray Diffraction (XRD)

XRD (X-ray Diffraction) is a device widely used in analysis. XRD uses X-rays to determine the crystal structure of a sample. X-rays are directed towards the sample and are reflected by atoms in the internal structure of the sample. These reflections, which rotate at a certain angle, reveal the crystal network structure, microstructures, phases, crystal dimensions and structure of the sample to be examined. At the same time, XRD can also examine microstructural features such as stress, strain and distortion in materials. An XRD analysis usually begins with obtaining a diffraction pattern. This pattern is then analyzed to provide information about the chemical composition, crystal structure and other microstructural features of the sample. XRD is widely used in the analysis of many different materials such as ceramics, metals, polymers, drugs and nanomaterials.

XRD analysis shows the elemental peaks of a sample. In this part of the thesis, XRD was used to verify the class of the titanium sample taken and in the later stages, it was used to detect the elemental peaks again after MAO. XRD analysis was carried out at Sakarya University Faculty of Engineering. Rigaku brand XRD device was used..The image of the XRD device used is given in Figure 5.24.



Figure 5.24 : Image of the XRD device.

5.11 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR (Fourier Transform Infrared) spectroscopy is an analysis method that provides information about the chemical structures of substances. FTIR starts by exposing the sample to infrared radiation and measures these interactions, where molecules interact with the radiation and cause vibrational movements. FTIR creates peaks that show absorption at certain wavelengths according to the properties of the chemical bonds of the molecule, and the interpretation of these peaks reveals the presence of functional groups in the sample.

This method is widely used in the structural analysis of organic compounds, polymers and biomolecules. FTIR is used in many areas such as chemical analysis, material characterization, environmental testing and pharmaceutical applications, and provides powerful, sensitive and unique results like the fingerprints of molecules. PerkinElmer brand FTIR device was used in this study.



6. CONCLUSIONS

6.1 Investigation of the SEM Images of Failed Implants

At this stage, unsuccessful implants that fell out of the mouth due to failure to achieve osseointegration were examined. For comparison purposes, SLA surface modification was performed on an unused implant. Figure 6.1, 6.2 and 6.3 shows the SEM images of unused SLA treated implant.

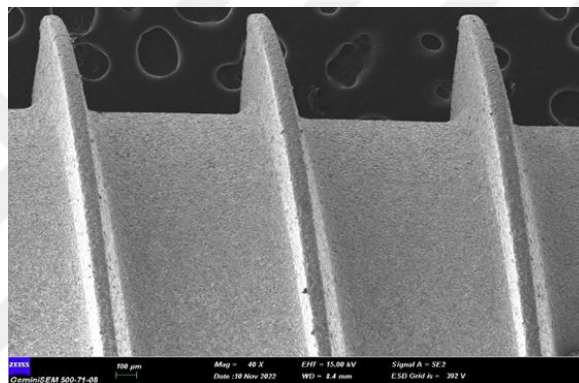


Figure 6.1 : SEM image of 4.3x13 mm implant.

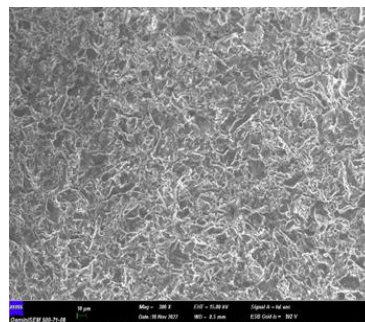


Figure 6.2 : Surface structure of the unused 4.3 x 13 mm implant.

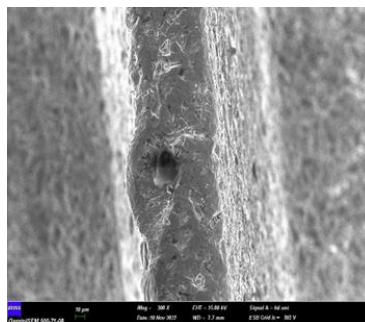


Figure 6.3 : Surface defect structure of the unused 4.3 x 13.

In the next step, implants that failed to achieve osseointegration and fell out were examined. The SEM image of the used implants under different magnifications is given in Figure 6.4, to 6.21.

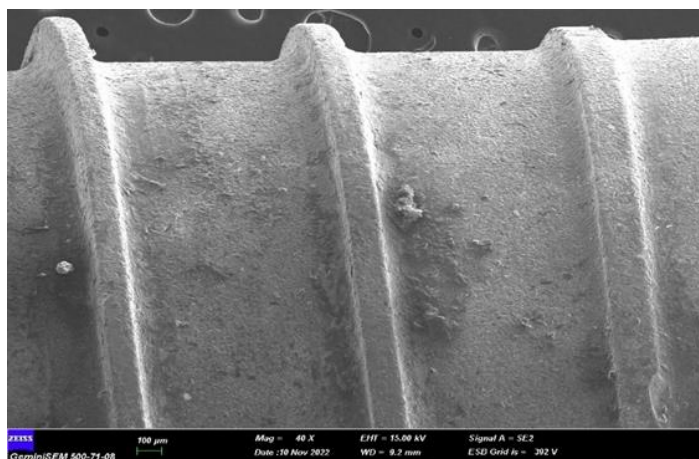


Figure 6.4 : SEM image of 3.4 x 13 mm implant.

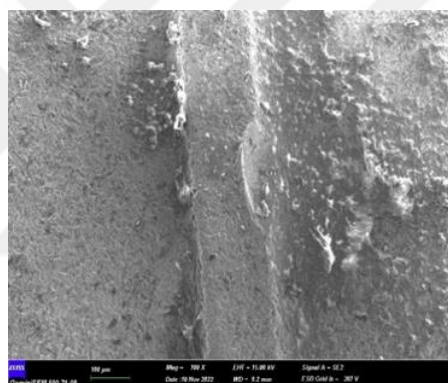


Figure 6.5 : Surface defect of 3.4 x 13 mm implant.

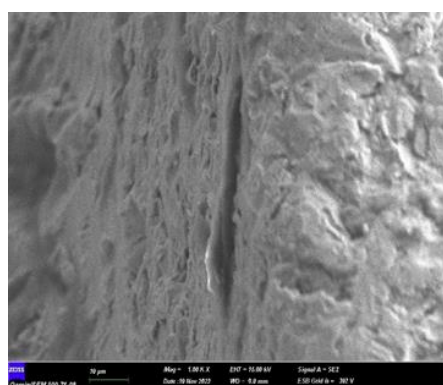


Figure 6.6 : 3.4 x 13 mm implant surface defect.

When examining Figure 6.5 and Figure 6.6 of the same implant, distinct grooves accompanying micro-roughness were observed on the surface. Micro-roughness is important for the adhesion of bone cells and osseointegration. However, residues and irregularities accumulated on the surface were observed here and there. It was thought

that contamination and surface defects could have a negative effect on the implant since they would affect its long-term performance. The crack-like lines and irregularities seen in Figure 6.5 suggest that corrosion may have weakened the surface structure. At the same time, a fracture-like structure was observed on the implant groove. Chewing forces may cause micro-cracks to form on the implant surface. This may lead to deterioration in the microstructure of the surface and weaken osseointegration. It was thought that the cracks and lines may be the result of such mechanical stresses.

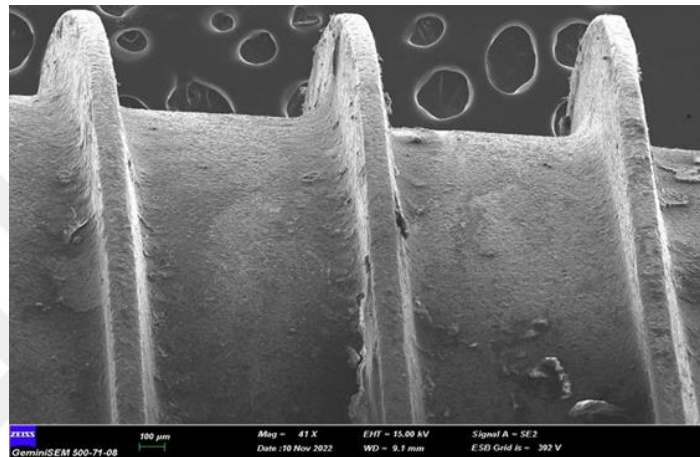


Figure 6.7 : SEM image of 4.3 x 11 mm implant.

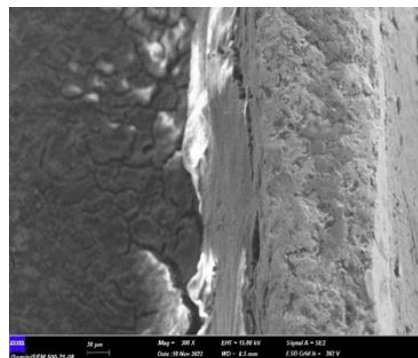


Figure 6.8 : Structure seen on the surface of 4.3 x 11 mm.

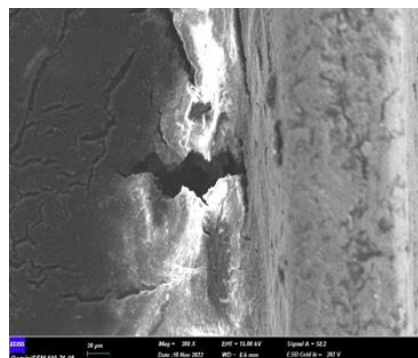


Figure 6.9 : Crack-like structure on 4.3 x 11 mm implant surface.

Images of the second implant were examined, micro-fracture and crack structures were observed on the surface. Uneven distribution of chewing forces after implant placement may lead to micro-cracks. It is thought that the surface cracks seen in Figure 6.8 and Figure 6.9 were formed due to mechanical loads or stress concentration. It is thought that these cracks may be the cause of failure as they may create entry points for corrosion and accelerate material fatigue.

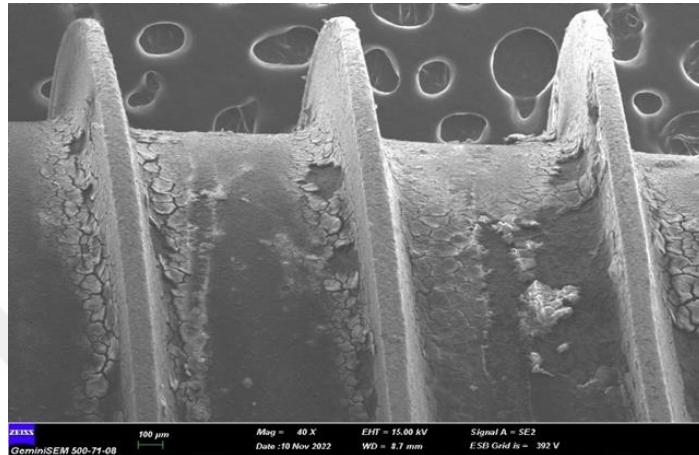


Figure 6.10 : SEM image of 4.3 x 13 mm implant.

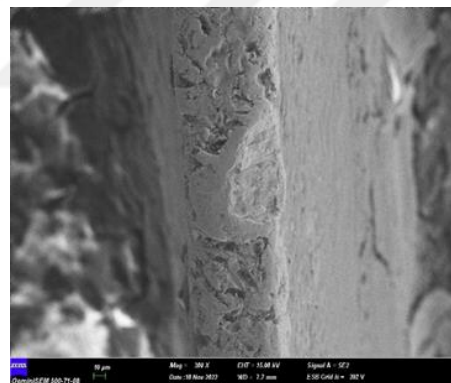


Figure 6.11 : Surface defect of 4.3 x 13 mm implant.

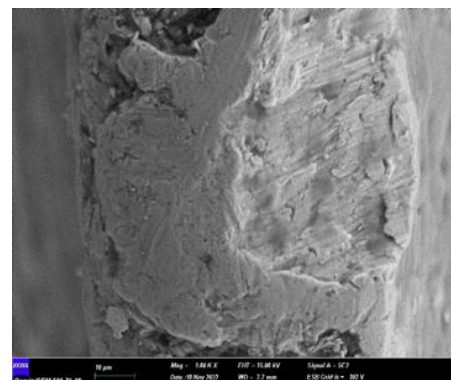


Figure 6.12 : Surface defect of 4.3 x 13 mm implant image at 1000x magnification.

When the images of the 3rd implant were examined, it was seen that the integrity of the coating was disrupted and showed an irregular distribution in Figure 6.10. Again, a surface fracture was observed, which was thought to be due to mechanical loading or stress concentration.

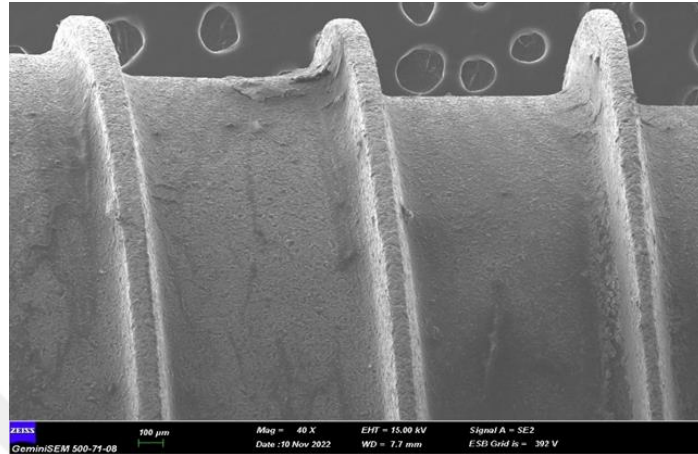


Figure 6.13 : SEM image of 4.3 x 9 mm implant.

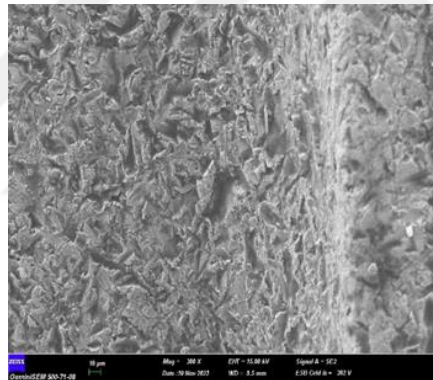


Figure 6.14 : Surface image of 4.3 x 9 mm implant.

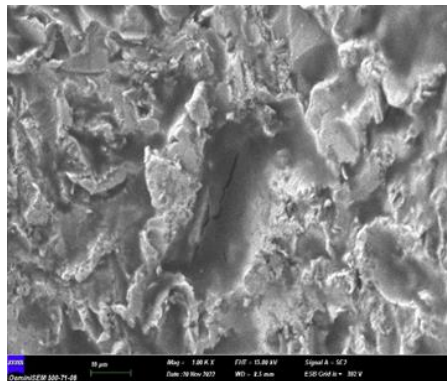


Figure 6.15 : Crack-like structure of 4.3 x 9 mm implant.

In the examination of the 4th implant surface, it is seen that the micro-rough structure is homogeneously distributed in Figure 6.13. A crack-like structure was seen among

the rough structures in Figure 6.14 and 6.15, thought to be caused by the damage during the sandblasting process.

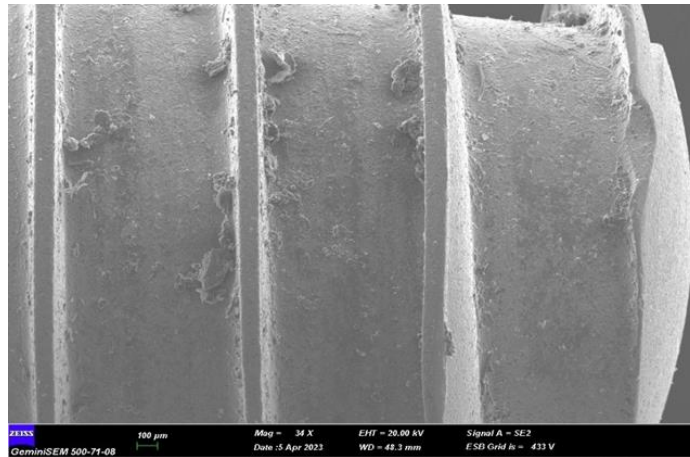


Figure 6.16 : SEM image of 4.3 x 7 mm implant.

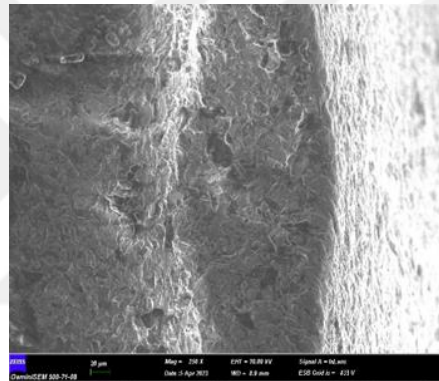


Figure 6.17 : Surface defect of 4.3 x 7 mm implant.

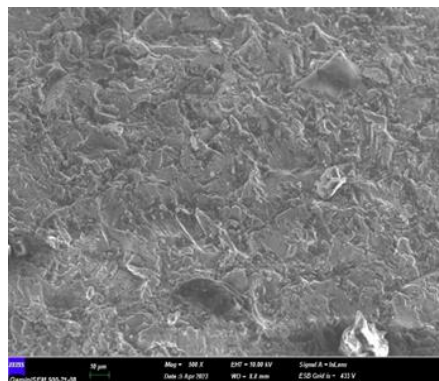


Figure 6.18 : Close-up view of surface defect.

In the examination of the 5th implant, fracture is observed at the apex of the dental implant. It is thought that excessive chewing or bruxism forces on the implant may cause stress accumulation in the apex region and may cause fracture. In addition, since the apex region may be overloaded when the load distribution is not adjusted properly as a result of improper occlusion, this possibility has also been considered. However,

the presence of sandblasted and rough structures on the fractured surface suggested that the implant fracture occurred before the implant placement procedure.

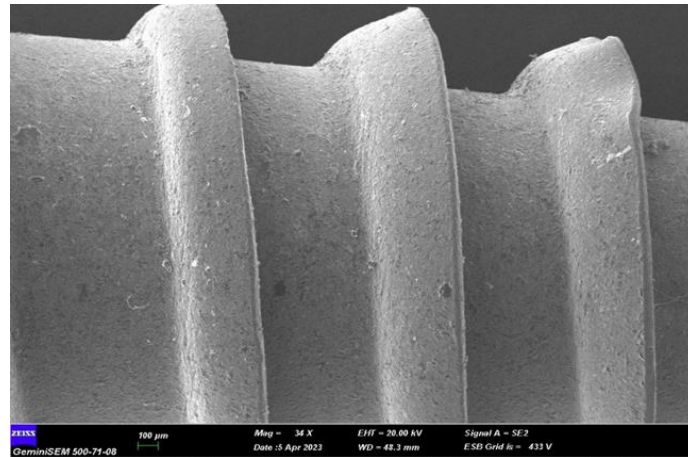


Figure 6.19 : SEM image of 3.4 x 11 mm implant.

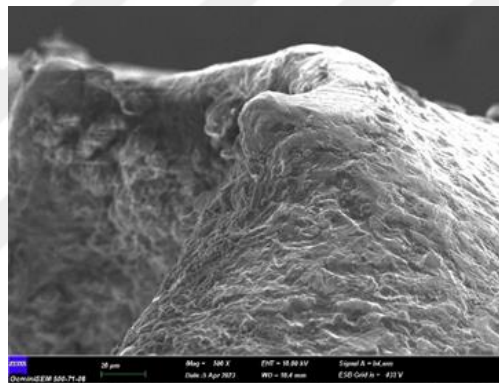


Figure 6.20 : Surface defect of 3.4 x 11 mm implant.

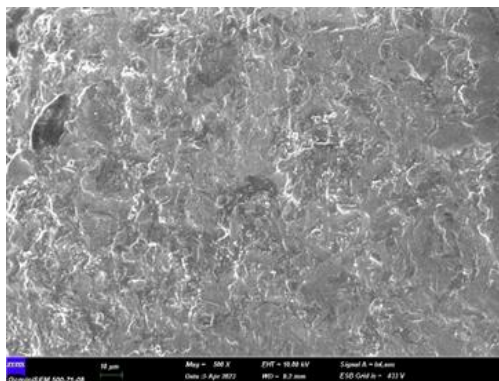


Figure 6.21 : Close-up view of defect.

In the examination of the 6th implant, serious deformation was observed in the thread close to the apex. It is thought that it is due to excessive chewing force on the implant or that the load distribution is not adjusted properly. In Figure 6.21, a grain thought to be aluminum oxide sand is seen. This grain is thought to be compatible with the shapes of the sand images taken.

6.2 Interpretation of Radiological Images

After SEM analysis, radiological images of implants were examined. It was observed that 10 implants examined did not contain fracture-like structures and had a holistic structure, and no discontinuity was observed. In Figure 6.22, implants numbered 1,2,3,4,5 in the AK section, are unused implants and were used for comparison. In reaching a conclusion, studies in the literature were examined and compared with radiological images of other implants. [237-238] Apex, body, groove and tip sections of implants were examined. Figure 6.23 and 6.24 show the close-up view of these implants.

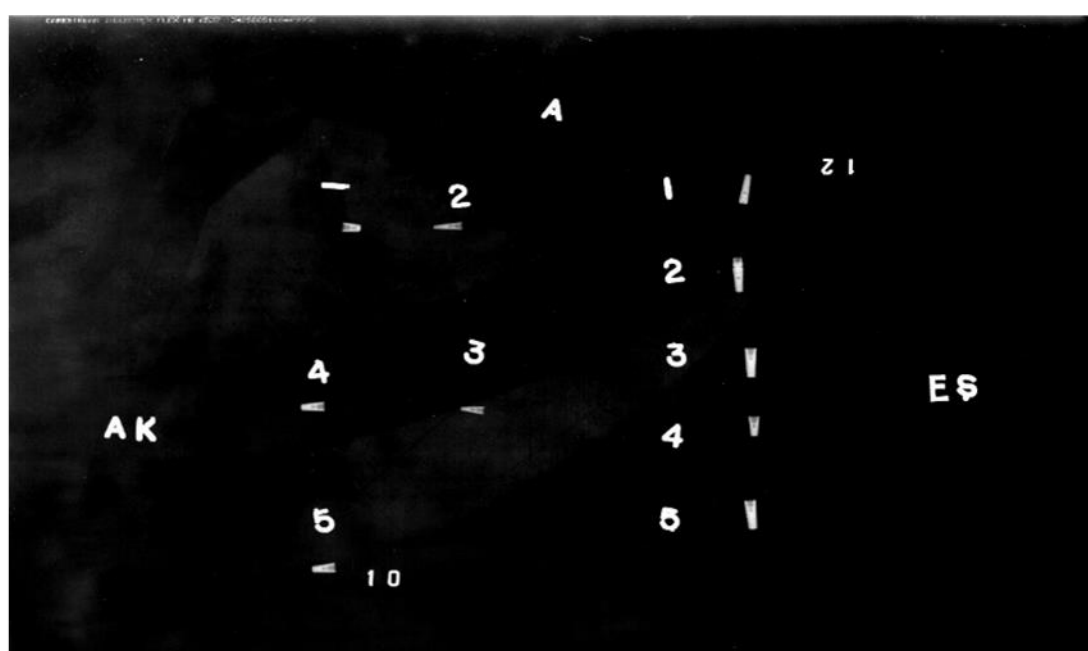


Figure 6.22 : Radiological images of unused implants (AK) and unsuccessful implants (ES).

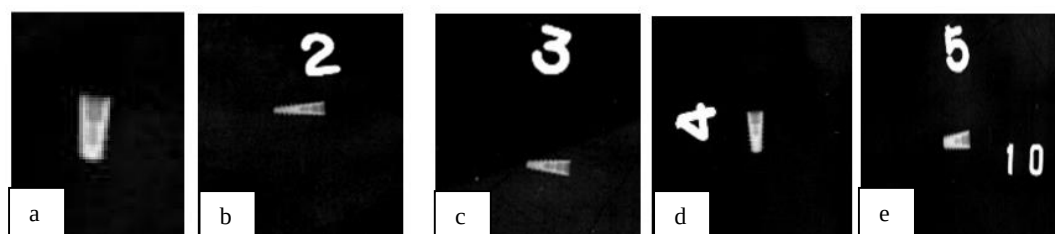


Figure 6.23 : Radiological image of a) 4.3 diameter and 7 mm long implant, b) 3.4 x 11 mm implant c) 3.4 x 9 mm implant d) 4.3 x 9 mm implant e) 4.3 x 9 mm implant.

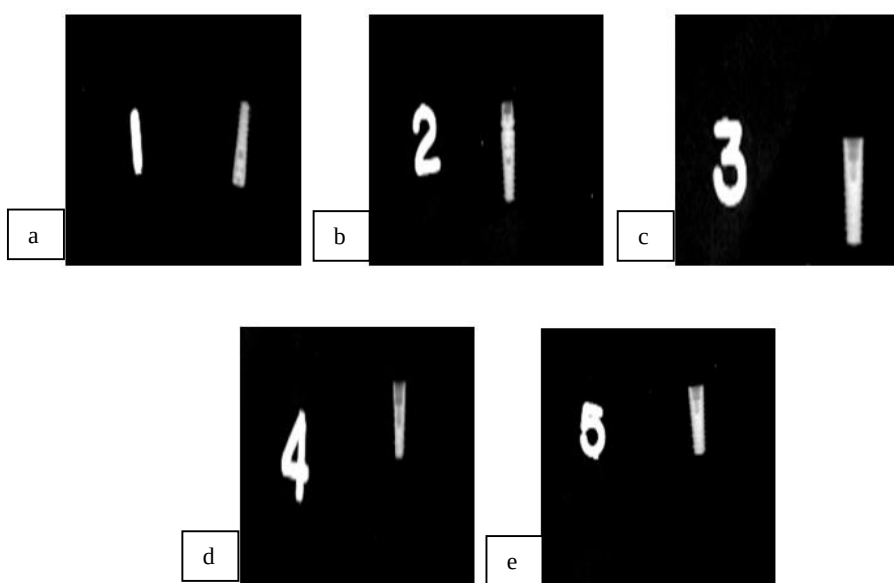


Figure 6.24 : Radiological image of a) 3.4 x 13 mm , b) 4.3 x 11 mm, c) 4.3 x 13 mm, d) 4.3 x 9 mm implant, e) 4.3 x 13 mm unsuccessful implant.

No significant deformity was noticed in these sections, but since radiological images were insufficient to visualize these sections, SEM images were considered more appropriate for a more accurate examination and evaluation of these structures.

6.3 SEM-EDS Analysis of Disc Samples

The Ti6Al4V titanium alloy implant used in the study was subjected to surface modifications using the SLA (Sandblasting & Acid Etching) method with aluminum oxide sands of different grit sizes. Osseointegration is the direct structural and functional connection between the bone tissue and the surface of the implant material and is known to be very important for the stability and success of dental implants. One of the most important parameters affecting osseointegration and therefore the success of dental implants is the surface properties of dental implants. The implant sandblasting process is based on the principle of spraying aluminum oxide sand grains onto the surface under a certain pressure and sandblasting the surface, and the subsequent acidification process is performed by removing residues on the surface after sandblasting and adding nano-roughness to the micro-rough surface.

Rough implants used in the body support bone growth and osseointegration since the bone-implant interface and surface roughness are directly related to the implant's success rate and biomechanical integration. SLA surface modification accelerates

bone integration and increases biocompatibility. The sizes of sand grains used for sandblasting are shown in Figures 6.25.

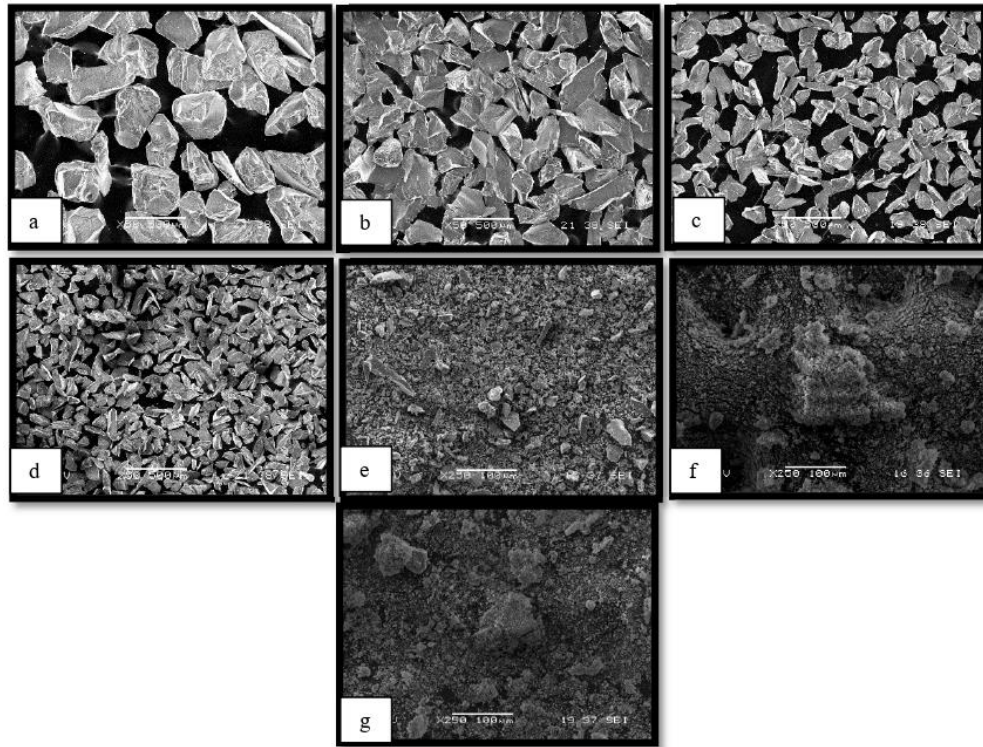


Figure 6.25 : a) Alumina 212-300 μm , b) 150-212 μm , c) 90-125 μm , d) 63-90 μm , e) 3-19 μm , f) 180 nm, g) 78 nm.

Due to its rough surface structure, it increases osseointegration, mechanical adhesion of the implant and increases clinical success. It is an effective surface modification method preferred in medical implants due to the advantages it offers and ease of application. Table 6.1 shows the abbreviated names of the samples used at this stage.

Table 6.1 : Abbreviated names of the samples.

Sample	Grit Size Used in Sandblasting	Acid Type	Abbreviation
1	212-300 μm	Citric Acid	F60-C
2	212-300 μm	Oxalic Acid	F60-O
3	150-212 μm	Citric Acid	F80-C
4	150-212 μm	Oxalic Acid	F80-O
5	90-125 μm	Citric Acid	F120-C
6	90-125 μm	Oxalic Acid	F120-O
7	60-93 μm	Citric Acid	F180-C
8	60-93 μm	Oxalic Acid	F180-O
9	3-19 μm	Citric Acid	F600-C
10	3-19 μm	Oxalic Acid	F600-O

SEM images after sandblasting and acid etching processes are given in the Figure 6.26 to Figure 6.27 and Figure 6.35 and Figure 6.36 shows the untreated sample.

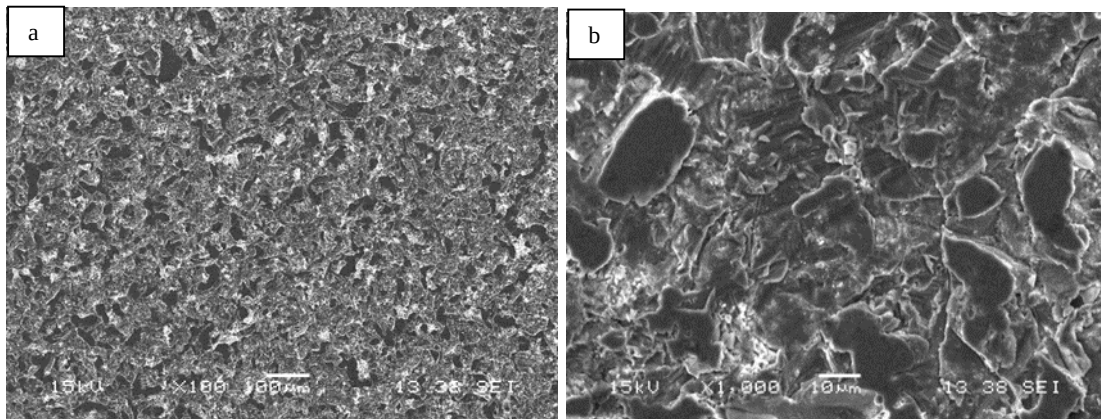


Figure 6.26 : SEM image of a) F60-C at 100x magnification, b) F60-C at 1000x magnification.

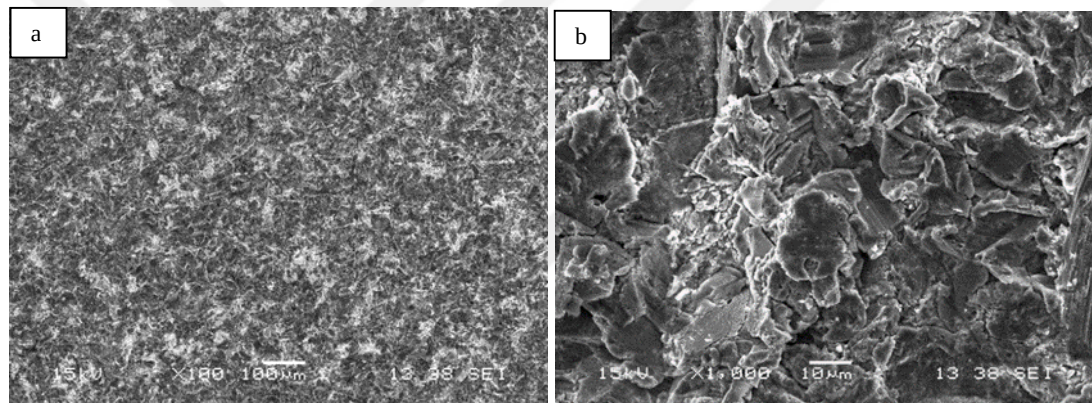


Figure 6.27 : SEM image of a) F60-O at 100x magnification, b) F60-O at 1000x magnification.

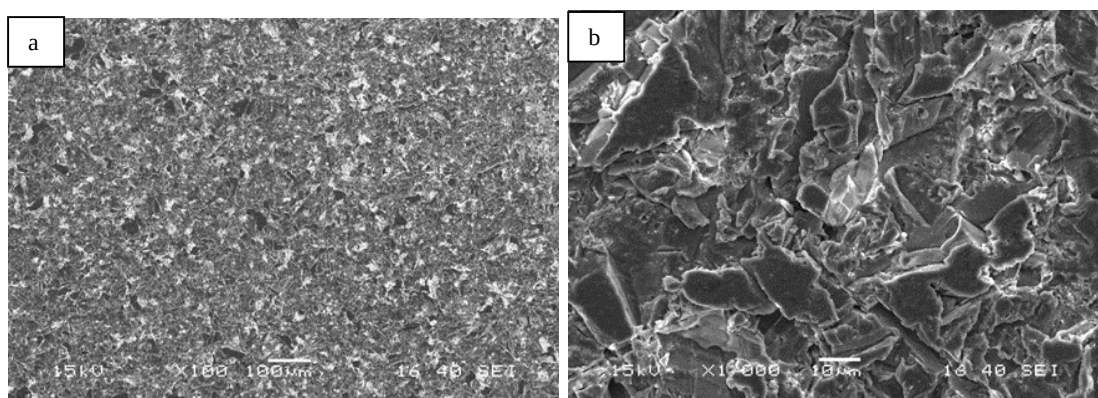


Figure 6.28 : SEM image of a) F80-C at 100x magnification, b) F80-C at 1000x magnification.

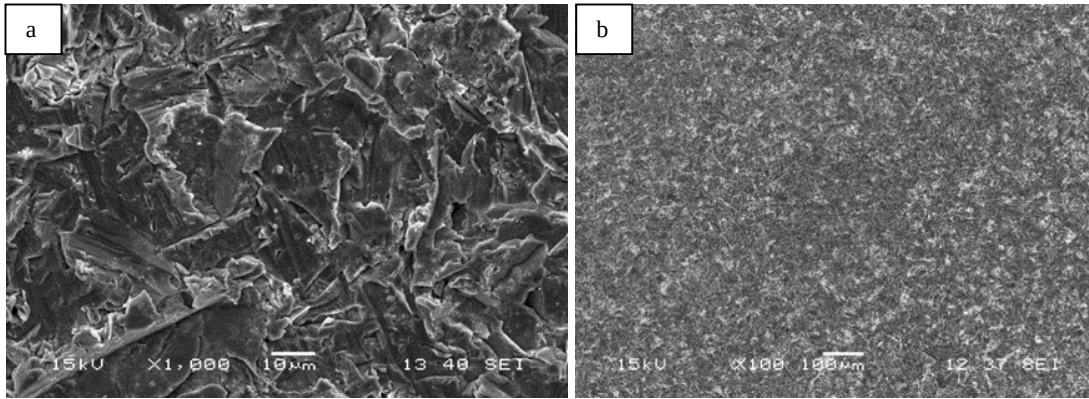


Figure 6.29 : SEM image of a) F80-O at 100x magnification, b) F80-O at 1000x magnification.

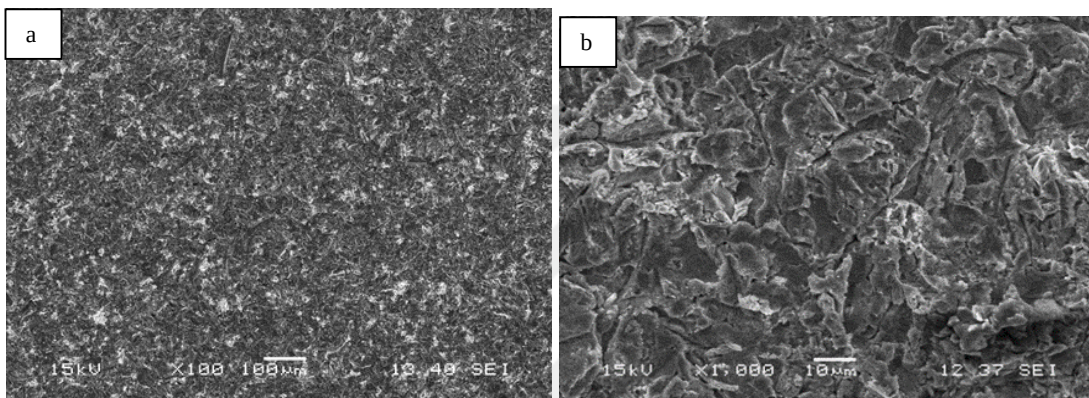


Figure 6.30 : SEM image of a) F120-C at 100x magnification, b) F120-C at 1000x magnification.

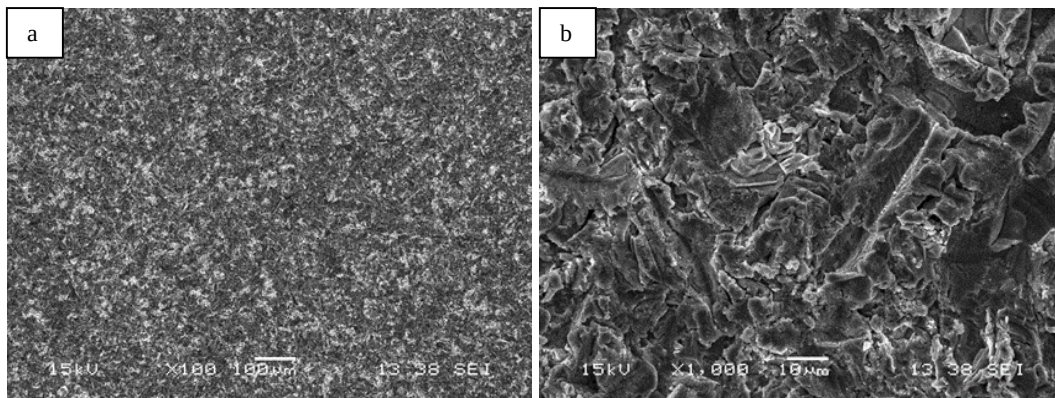


Figure 6.31 : SEM image of a) F120-O at 100x magnification, b) F120-O at 1000x magnification.

A rough surface is observed as a result of SLA. It is seen that there are more indented structures in the samples where large grained sands are used.

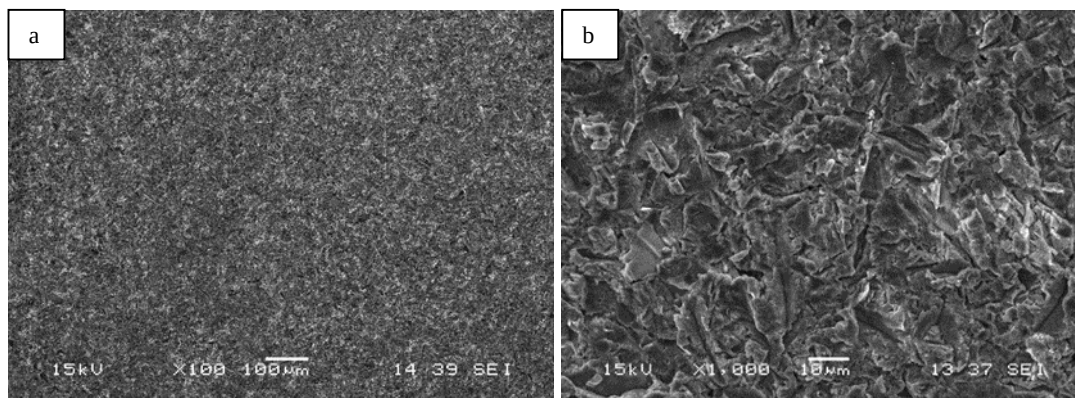


Figure 6.32 : SEM image of a) F180-C at 100x magnification, b) F180-C at 1000x magnification.

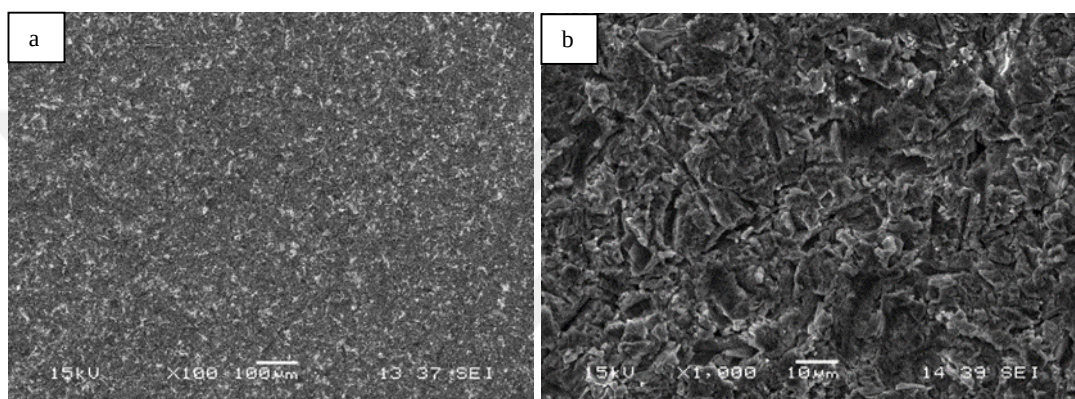


Figure 6.33 : SEM image of a) F180-O at 100x magnification, b) F180-O at 1000x magnification.

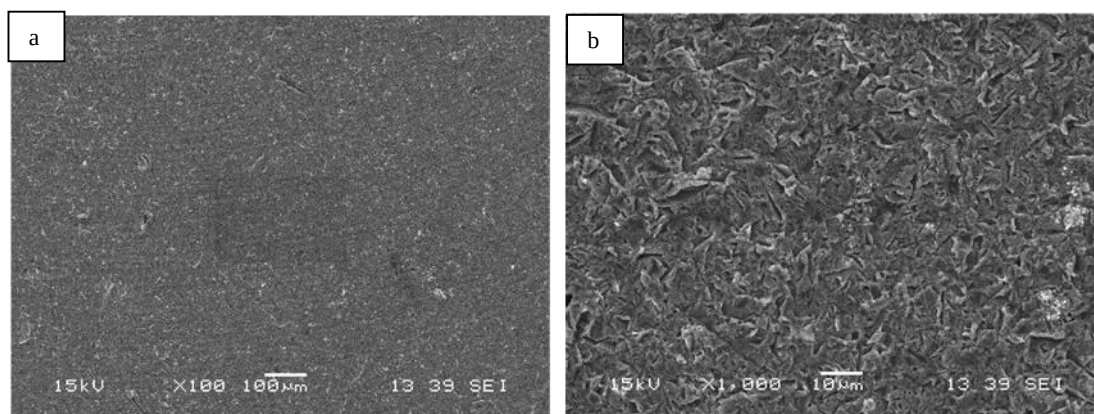


Figure 6.34 : SEM image of a) F600-C at 100x magnification, b) F600-C at 1000x magnification.

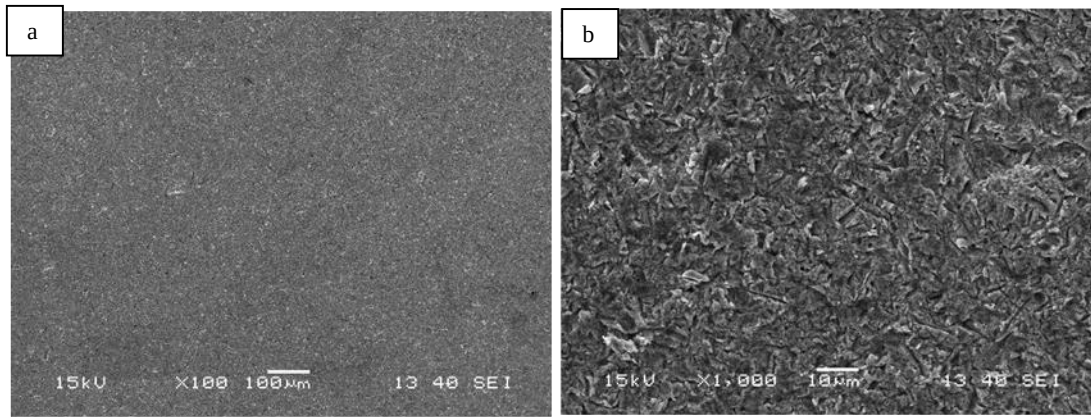


Figure 6.35 : SEM image of a) F600-O at 100x magnification, b) F600-O at 1000 x magnification.

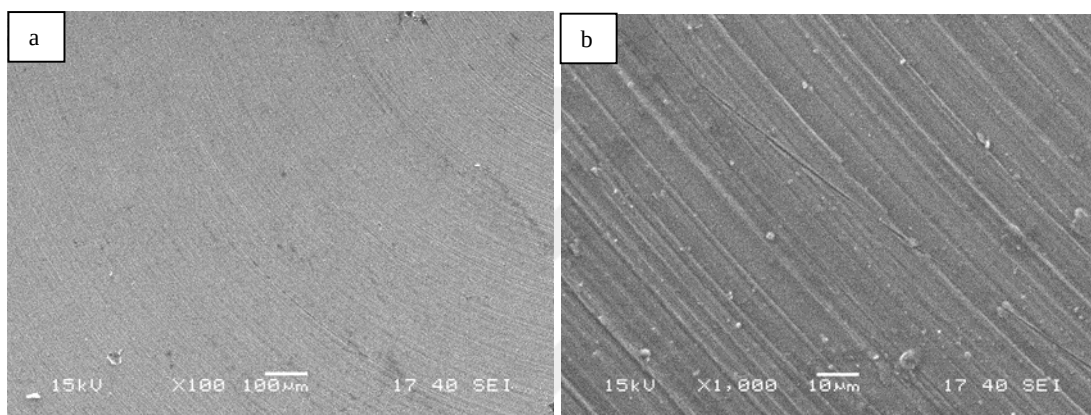
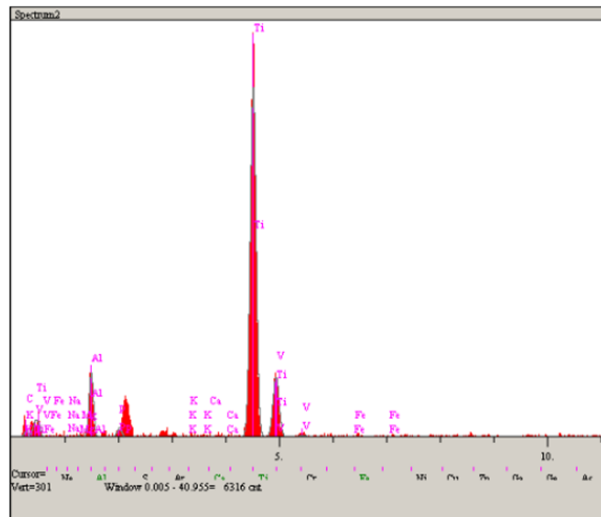


Figure 6.36 : SEM image of a) untreated surface at 100x magnification, b) untreated surface at 1000x magnification.

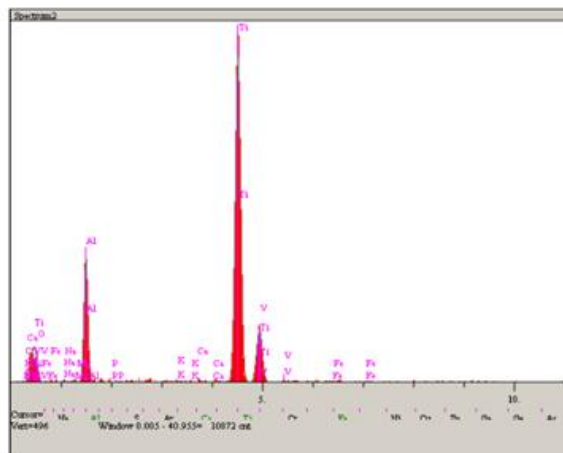
Compared to the untreated surface, it is seen that high roughness is obtained on the surfaces of implants and disks that have undergone sandblasting and acid etching processes. The rough surfaces obtained as a result of the process can be seen from the contrasts and depths of field in SEM images. It was observed that the depths of field decrease with the decreasing sand sizes. The results of EDS analysis performed on the surface of the disk samples are given below. According to the analysis of the untreated surface, the obtained results were measured as 85.2 wt.% Titanium, 3.9 wt.% Vanadium, 7.2 wt.% Aluminum. EDX results of untreated disk sample is given in Figure 6.37.

The results obtained from the treated; sandblasted and then acid etched surfaces are given in Figure 6.38 to 6.47.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	4.00	1.265	1.810	wt. %	
O	Ka	0.00	0.000	0.000	wt. %	
Na	Ka	0.57	0.476	0.138	wt. %	
Mg	Ka	0.87	0.588	0.151	wt. %	
Al	Ka	46.65	4.319	7.241	wt. %	
P	Ka	7.19	1.696	1.071	wt. %	
K	Ka	0.79	0.563	0.116	wt. %	
Ca	Ka	0.29	0.343	0.042	wt. %	
Ti	Ka	346.41	11.770	85.201	wt. %	
V	Ka	13.73	2.343	3.896	wt. %	
Fe	Ka	0.67	0.517	0.333	wt. %	
				100.000	wt. %	Total

Figure 6.37 : EDS results of untreated disk sample.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	0.00	0.000	0.000	wt. %	
O	Ka	8.08	1.797	5.480	wt. %	
Na	Ka	1.88	0.866	0.227	wt. %	
Mg	Ka	2.68	1.034	0.234	wt. %	
Al	Ka	167.66	8.188	13.13	wt. %	
P	Ka	0.28	0.333	0.022	wt. %	
K	Ka	2.65	1.028	0.208	wt. %	
Ca	Ka	1.50	0.775	0.117	wt. %	
Ti	Ka	610.34	15.62	77.33	wt. %	
V	Ka	16.98	2.606	2.482	wt. %	
Fe	Ka	3.00	1.096	0.764	wt. %	
				100.000	wt. %	Total

Figure 6.38 : EDS results of F60-C.

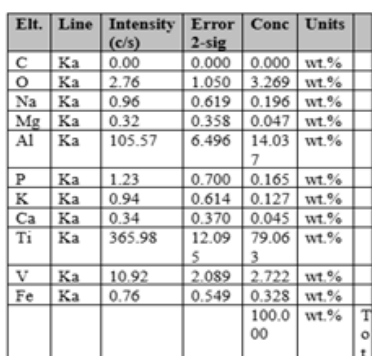


Figure 6.39 : EDS results of F60-O.

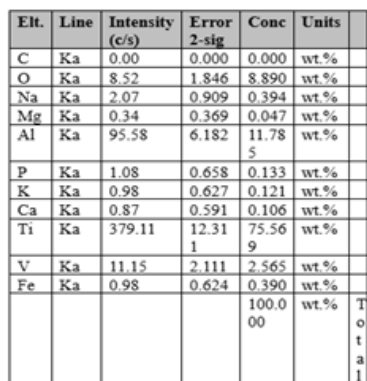
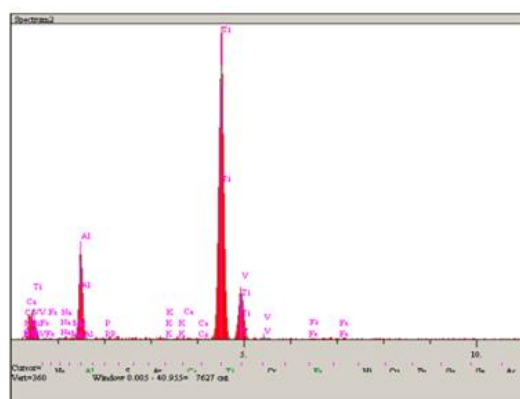
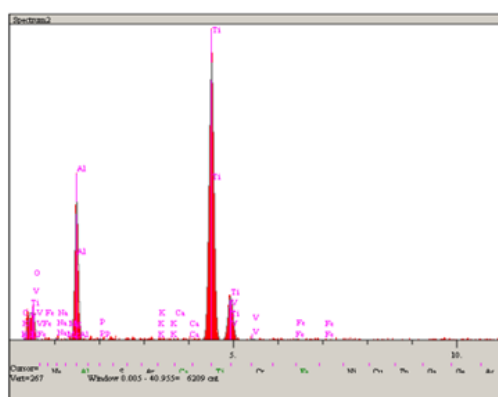


Figure 6.40 : EDS results of F80-C.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units
C	Ka	0.00	0.000	0.000	wt. %
O	Ka	9.98	1.998	9.286	wt. %
Na	Ka	1.73	0.831	0.295	wt. %
Mg	Ka	0.78	0.560	0.097	wt. %
Al	Ka	97.07	6.229	10.63	wt. %
P	Ka	0.89	0.597	0.096	wt. %
K	Ka	1.10	0.665	0.120	wt. %
Ca	Ka	1.23	0.702	0.132	wt. %
Ti	Ka	428.45	13.08	75.50	wt. %
V	Ka	16.84	2.595	3.427	wt. %
Fe	Ka	1.16	0.680	0.410	wt. %
				100.00	Total

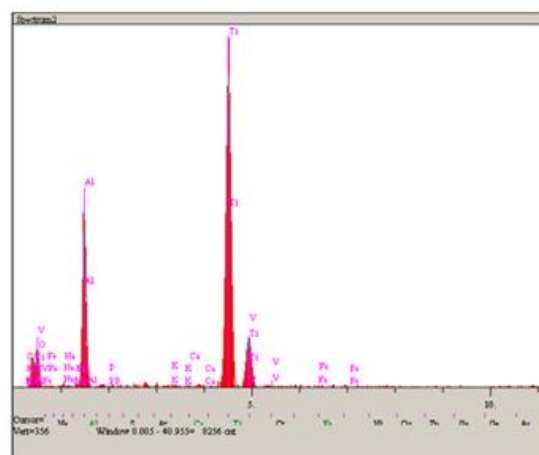
Figure 6.41 : EDS results of F80-O.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units
C	Ka	0.00	0.000	0.000	wt. %
O	Ka	17.33	2.633	16.36	wt. %
Na	Ka	1.74	0.833	0.332	wt. %
Mg	Ka	0.90	0.599	0.125	wt. %
Al	Ka	116.40	6.823	14.50	wt. %
P	Ka	1.06	0.650	0.134	wt. %
K	Ka	0.31	0.353	0.041	wt. %
Ca	Ka	0.97	0.622	0.126	wt. %
Ti	Ka	321.66	11.34	66.09	wt. %
V	Ka	7.36	1.716	1.747	wt. %
Fe	Ka	1.30	0.721	0.533	wt. %
				100.00	Total

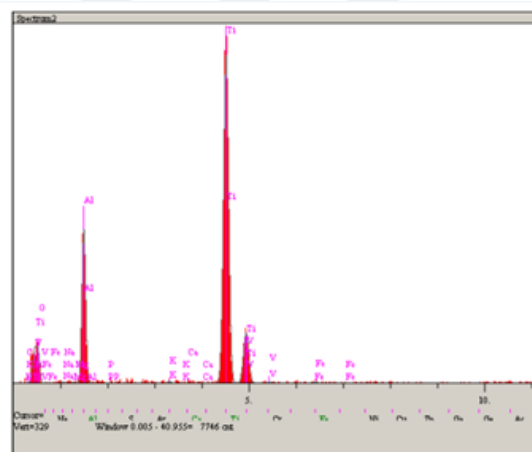
Figure 6.42 : EDS results of F120-C.

The main components are Ti, Al and V, which is consistent with the Ti-6Al-4V alloy. The oxygen content indicates oxidation or TiO₂ layer formation on the surface, while traces of Ca and P may indicate apatite formation in terms of biological activity.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units
C	Ka	0.00	0.000	0.000	wt. %
O	Ka	16.45	2.565	12.710	wt. %
Na	Ka	2.68	1.035	0.401	wt. %
Mg	Ka	0.28	0.337	0.031	wt. %
Al	Ka	164.03	8.098	16.046	wt. %
P	Ka	1.00	0.634	0.102	wt. %
K	Ka	1.98	0.890	0.205	wt. %
Ca	Ka	0.96	0.620	0.099	wt. %
Ti	Ka	419.97	12.957	68.063	wt. %
V	Ka	11.12	2.108	2.079	wt. %
Fe	Ka	0.82	0.572	0.265	wt. %
				100.000	Total

Figure 6.43 : EDS results of F120-O.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units
C	Ka	0.00	0.000	0.000	wt. %
O	Ka	11.39	2.134	9.961	wt. %
Na	Ka	1.28	0.714	0.210	wt. %
Mg	Ka	1.00	0.634	0.120	wt. %
Al	Ka	139.60	7.471	14.950	wt. %
P	Ka	0.09	0.195	0.010	wt. %
K	Ka	1.91	0.874	0.212	wt. %
Ca	Ka	0.55	0.470	0.061	wt. %
Ti	Ka	410.64	12.814	72.152	wt. %
V	Ka	8.05	1.795	1.633	wt. %
Fe	Ka	1.97	0.888	0.692	wt. %
				100.000	Total

Figure 6.44 : EDS results of F180-C.

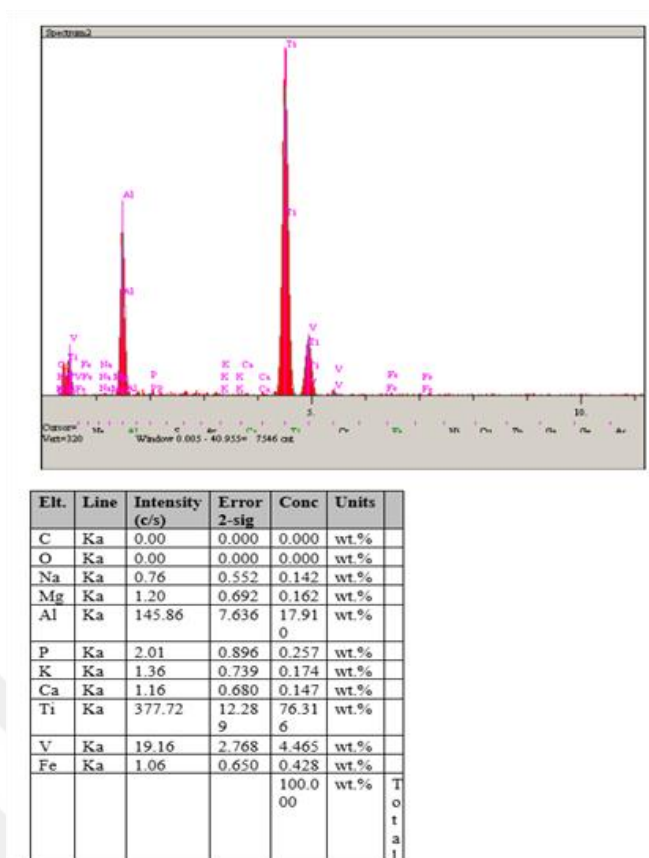


Figure 6.45 : EDS results of F180-O.

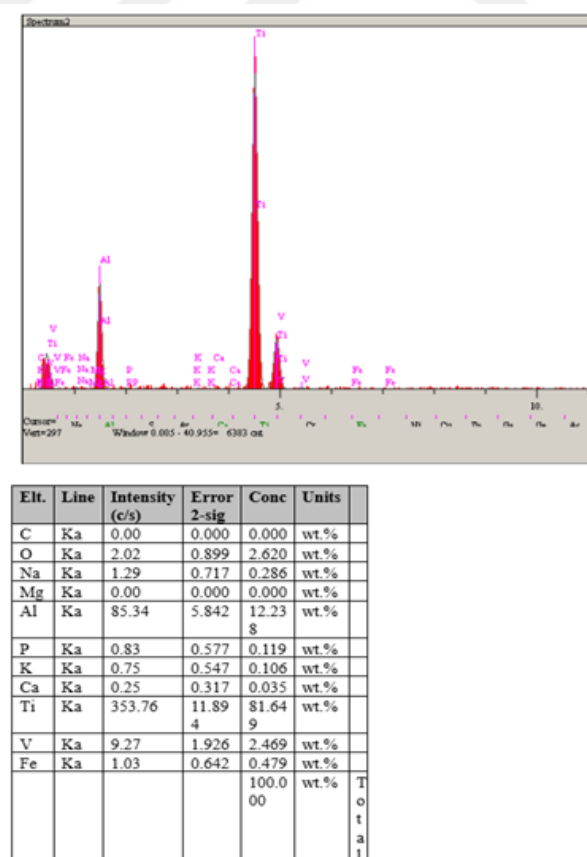


Figure 6.46 : EDS results of F600-C.

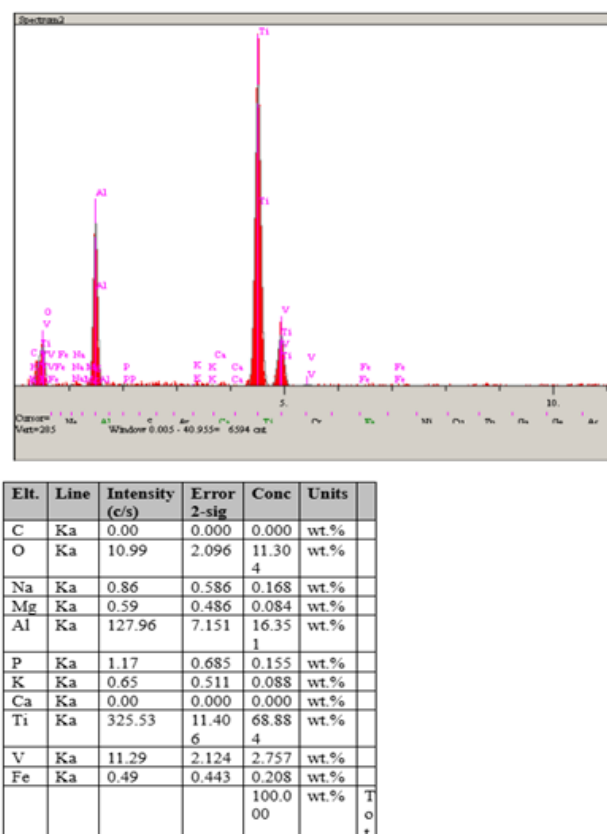


Figure 6.47 : EDS results of F600-O.

In the data obtained as a result of EDS analyses, no compound originating from the sandblasting method or sandblasting device was found. It is thought that the carbon seen in the untreated sample and the magnesium element in the other samples originated from sandblasting material and organic substances such as fingerprints, dirt or oil. It is thought that the oxygen found in the EDS analysis is present as a part of the aluminum oxide compound. It is thought that the sodium originated from the titanium production process. It is thought that the iron originated from the micro particles that broke off from the tip during the cutting of the titanium discs, while the signals of elements such as phosphorus, calcium and potassium were thought to be formed due to the contamination formed during the disc cutting.

6.4 XRD Analysis of Untreated Sample

XRD analysis was applied to the surface of the untreated disk sample and the results are presented in Figure 6.48.

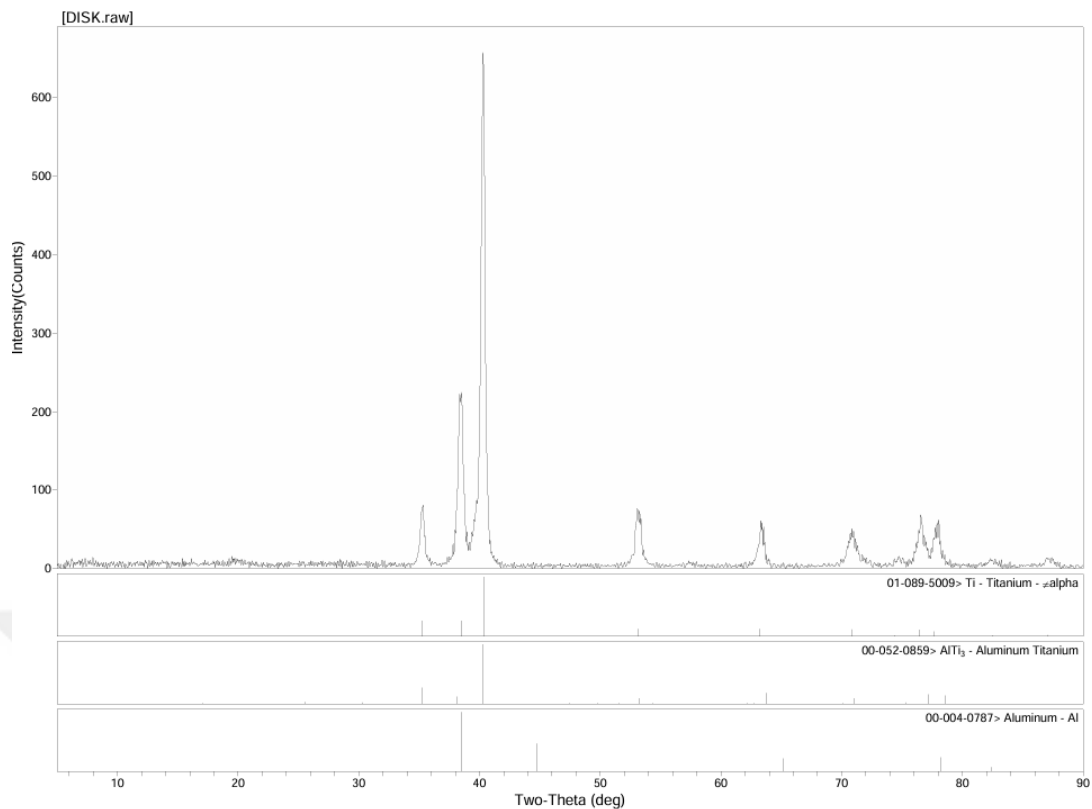


Figure 6.48 : XRD analysis of untreated disc.

XRD analysis was applied to the surface of the untreated disc sample and the results are presented as follows. Ti (α), AlTi₃-Aluminum Titanium and Aluminum-Al were found in the XRD analysis results. According to the obtained result, it shows that titanium is in the alpha phase in the material and there are AlTi₃ and pure aluminum phases. Although the material is a Ti6Al4V titanium alloy, there is no peak belonging to Vanadium or β -phase in the pattern. The reason why the Vanadium peak, which is a β -stabilizer in the Ti6Al4V titanium alloy, could not be detected in the XRD analysis can be explained by the limitations of XRD in detecting elements with low atomic numbers.

Vanadium peak can be seen in the XRD patterns of Ti6Al4V produced using heat treatment conditions or additive methods. Bartłomiej et al. observed Ti (β) in the patterns of the alloy produced by SLM (selective laser melting) and Electron Beam Melting (EBM) [229].

6.5 SEM Analysis of SLA Treated Samples After SBF

After rinsing the samples removed from the SBF with pure water, EDS-SEM analyses were performed. Figure 6.49 shows the surface images of the implant that was sandblasted with 3-19 μm sand and then etched and Figure 6.50 shows the EDS result.

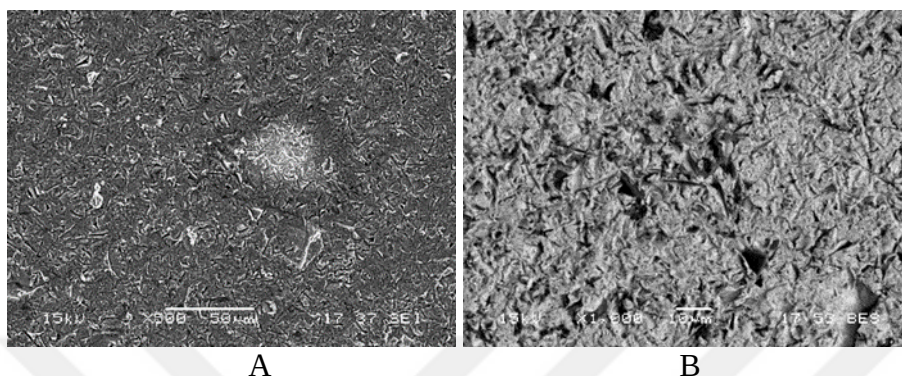
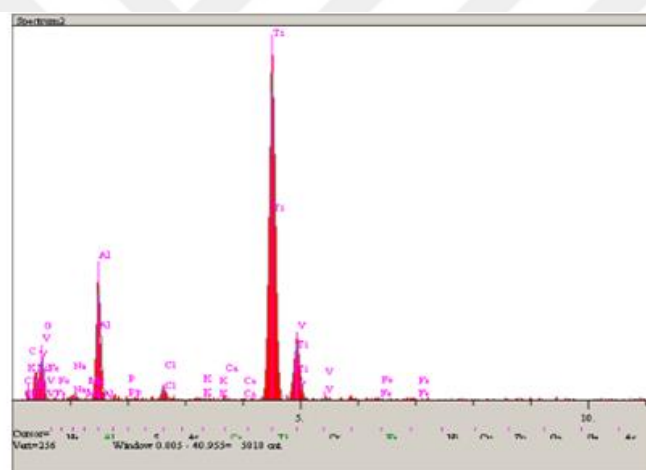


Figure 6.49 : A, B F600-C 500x and 1000x magnified.



El.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
C	Ka	0.00	0.000	0.000	wt.%	
O	Ka	6.09	1.560	7.626	wt.%	
Na	Ka	3.75	1.225	0.857	wt.%	
Mg	Ka	0.91	0.605	0.152	wt.%	
Al	Ka	79.01	5.620	11.729	wt.%	
P	Ka	0.97	0.624	0.144	wt.%	
Cl	Ka	9.71	1.970	1.413	wt.%	
K	Ka	1.59	0.797	0.241	wt.%	
Ca	Ka	1.42	0.752	0.213	wt.%	
Ti	Ka	306.37	11.068	73.634	wt.%	
V	Ka	11.77	2.169	3.263	wt.%	
Fe	Ka	1.51	0.778	0.729	wt.%	
				100.000	wt.%	Tot

Figure 6.50 : EDS analysis of F600-C.

Figure 6.51 shows the surface images of the implant that was sandblasted with 90-125 μm sandblasted and then citric acid etched and Figure 6.52 shows the EDS result of this implant.

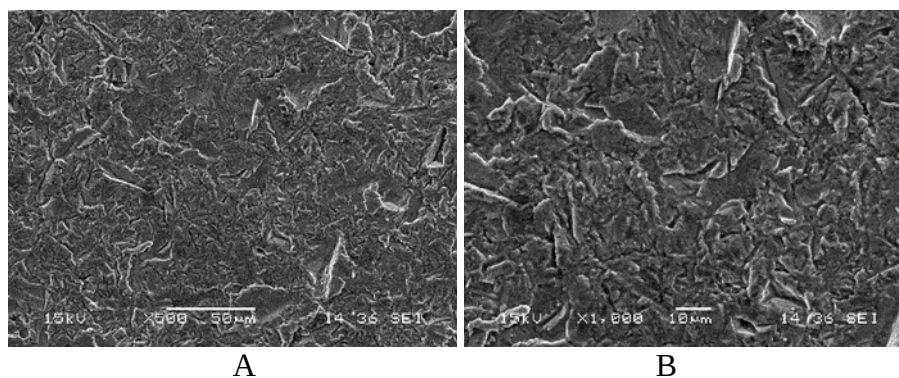
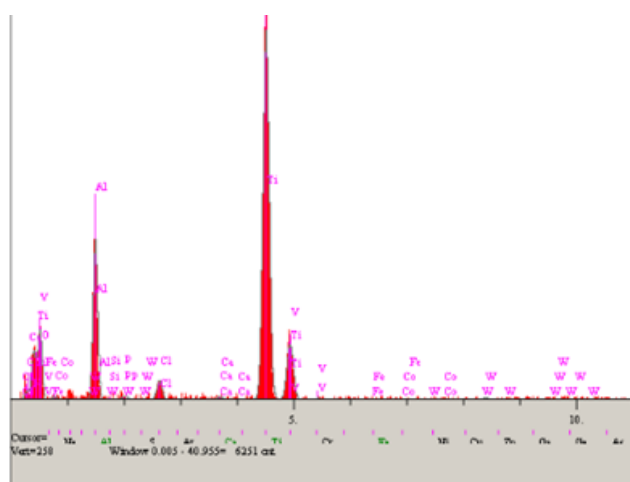


Figure 6.51 : A, B F120-C 500x and 1000x magnified.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units
C	Ka	4.80	1.385	2.181	wt. %
N	Ka	22.45	2.995	10.45	wt. %
O	Ka	0.00	0.000	0.000	wt. %
Al	Ka	95.45	6.175	12.26	wt. %
Si	Ka	0.37	0.387	0.049	wt. %
P	Ka	2.59	1.017	0.348	wt. %
Cl	Ka	12.96	2.276	1.743	wt. %
Ca	Ka	2.16	0.929	0.306	wt. %
Ti	Ka	302.93	11.00	66.18	wt. %
V	Ka	5.30	1.455	1.333	wt. %
Fe	Ka	0.74	0.543	0.319	wt. %

Figure 6.52 : EDS analysis of F120-C.

Figure 6.53 shows the surface images of the implant that was sandblasted with 212-300 μm sandblasted and then citric acid etched and Figure 6.54 shows the EDS results of this implant.

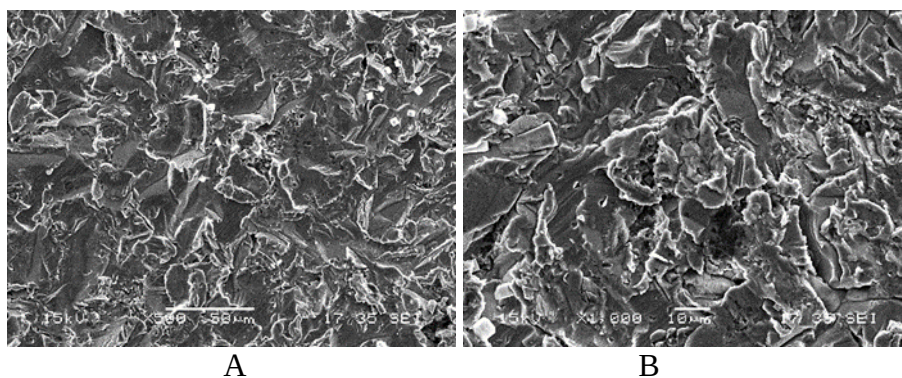
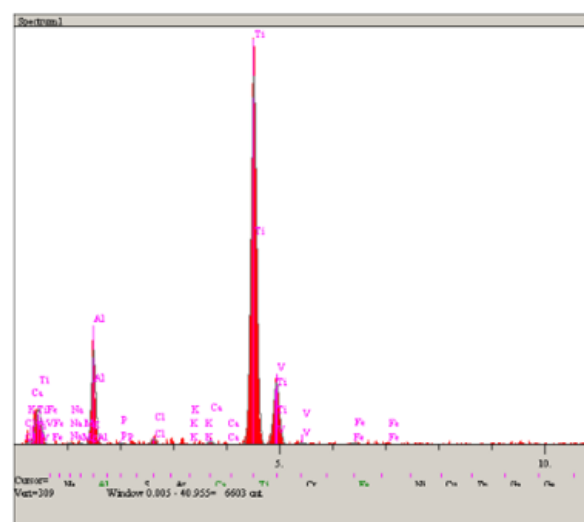


Figure 6.53 : A, B F60-C 500x and 1000x magnified.



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units
C	Ka	1.93	0.879	0.756	wt. %
O	Ka	6.81	1.650	7.587	wt. %
Na	Ka	1.33	0.729	0.267	wt. %
Mg	Ka	0.98	0.625	0.141	wt. %
Al	Ka	69.81	5.283	8.994	wt. %
P	Ka	1.43	0.755	0.179	wt. %
Cl	Ka	5.34	1.461	0.660	wt. %
K	Ka	0.42	0.409	0.053	wt. %
Ca	Ka	2.95	1.087	0.368	wt. %
Ti	Ka	373.92	12.22	77.10	wt. %
			6	8	
V	Ka	13.04	2.283	3.103	wt. %
Fe	Ka	1.89	0.869	0.784	wt. %

Figure 6.54 : EDS analysis of F60-C.

When the specimens examined under SEM, apatite structures exhibit certain micromorphological features. SEM images may vary depending on the preparation method and size of the sample, but apatite structures generally show clusters and aggregates. [230-231] It was observed that no such structures were formed in the examined SEM-EDS analysis images.

6.6 SEM and EDS Analysis of Samples After MAO Surface Treatment

After the samples removed from the SBF were rinsed with appropriate deionized water and dried at room temperature, SEM-EDS analysis was performed again. Figure 6.55, 6.56 shows the SEM and EDS images of the sandblasted with 212-300 μm Alumina sand and oxalic acid etched sample.

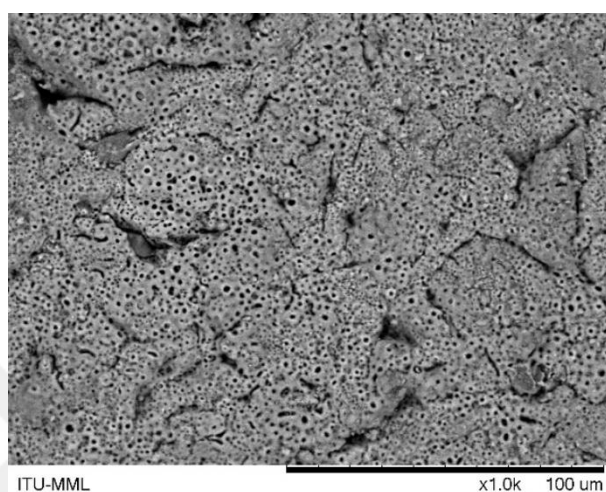


Figure 6.55 : F60-O disc SEM image.

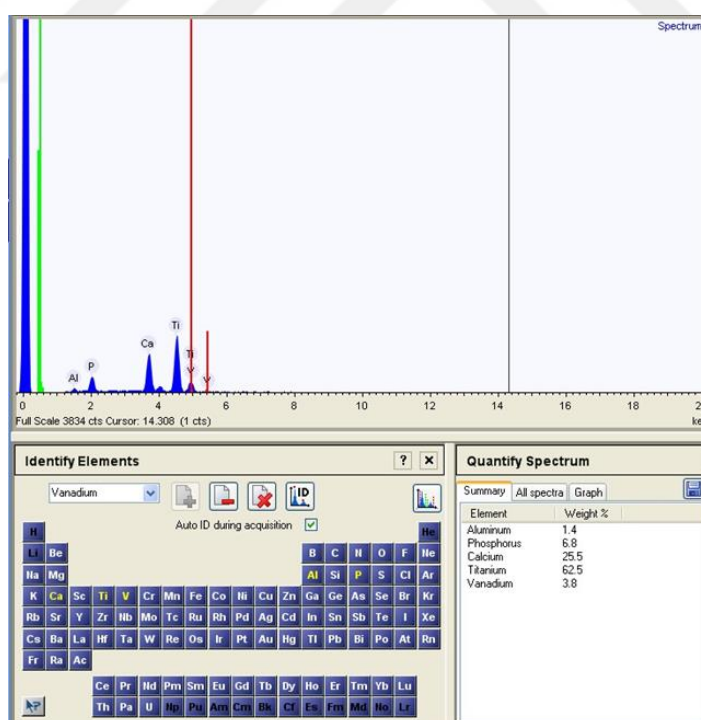


Figure 6.56 : EDS analysis of F60-O disc.

Figure 6.57, 6.58 shows the SEM and EDS images of the sandblasted with 150-212 μm Alumina sand and citric acid etched sample.

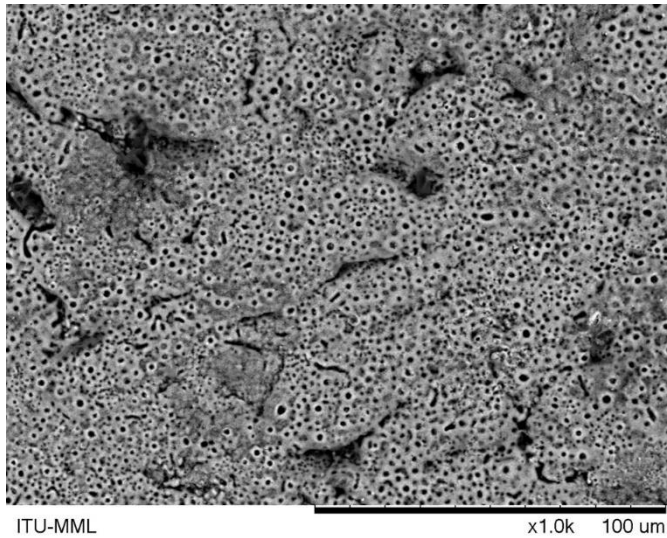


Figure 6.57 : F80-C disc SEM image.

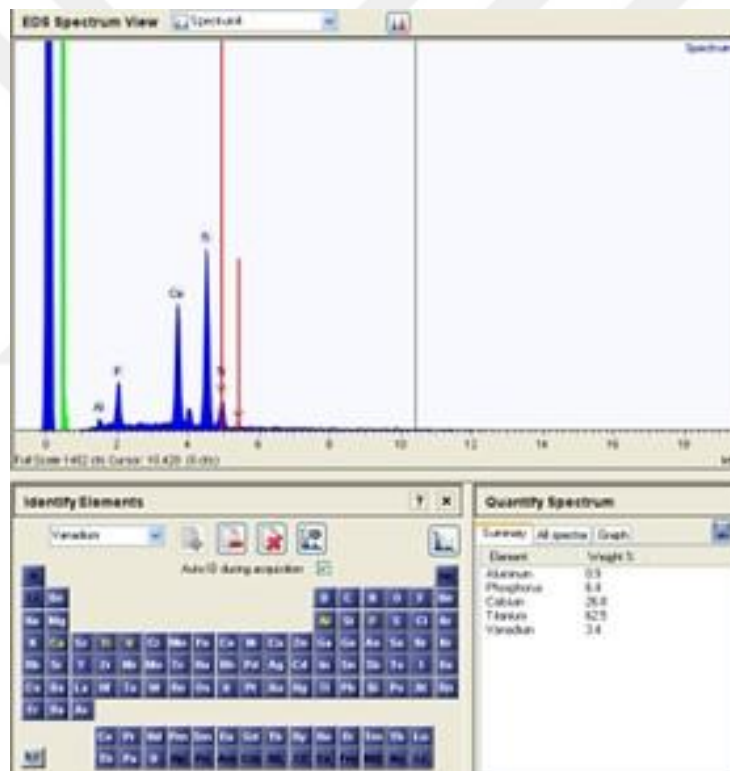


Figure 6.58 : EDS analysis of F80-C disc.

Figure 6.59, 6.60 shows the SEM and EDS images of the sandblasted with 90-125 μm Alumina sand and oxalic acid etched sample.

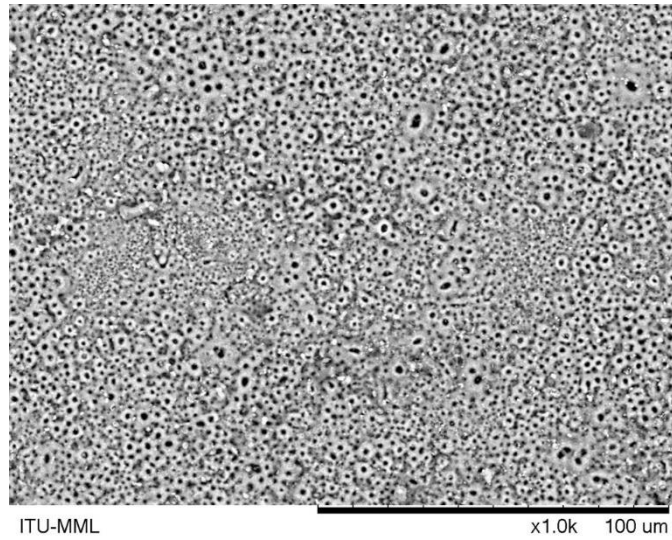


Figure 6.59 : F120-O disc SEM image.

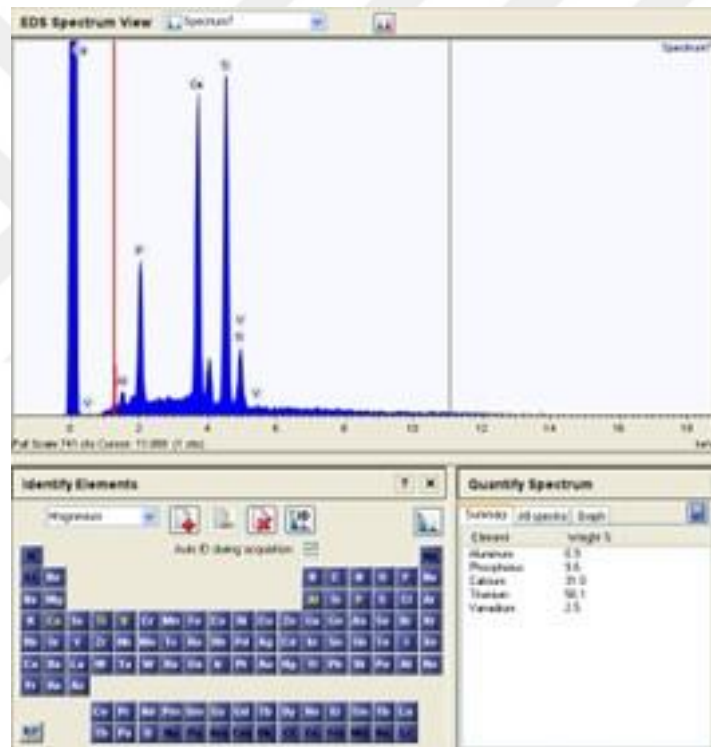


Figure 6.60 : EDS analysis of F120-O disc.

Figure 6.61, 6.62 shows the SEM and EDS images of the sandblasted with 63-90 μm Alumina sand and citric acid etched sample.

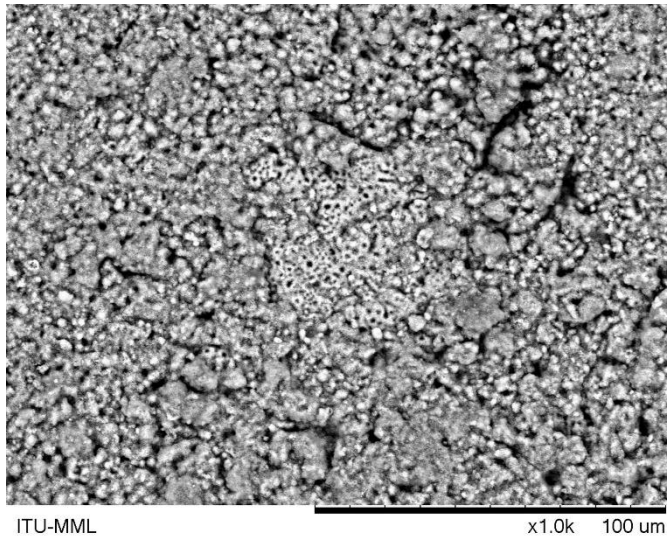


Figure 6.61 : F180-C disc SEM image.

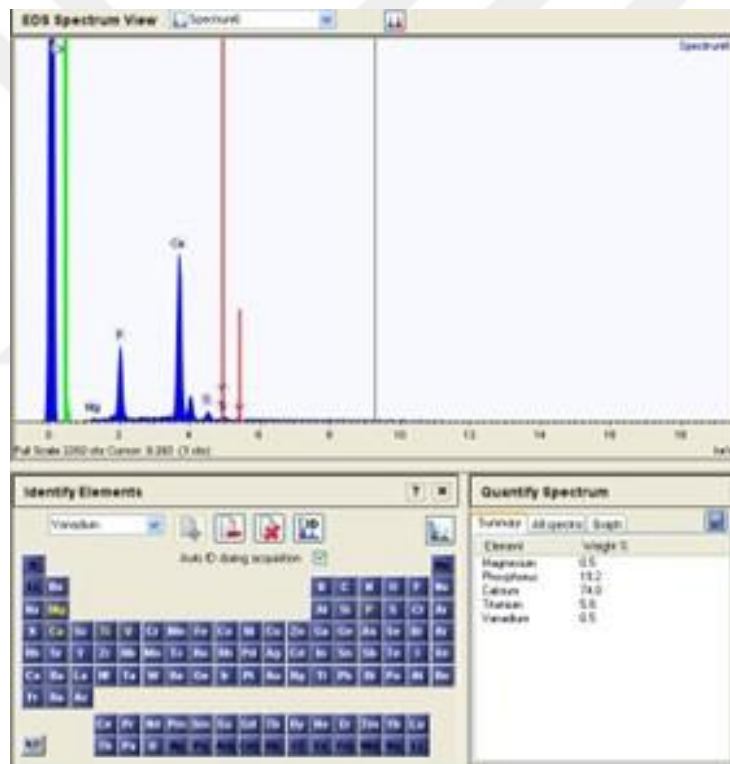


Figure 6.62 : EDS analysis of F180-C disc.

Figure 6.63, 6.64 shows the SEM and EDS images of the sandblasted with 3-19 μm Alumina sand and oxalic acid etched sample.

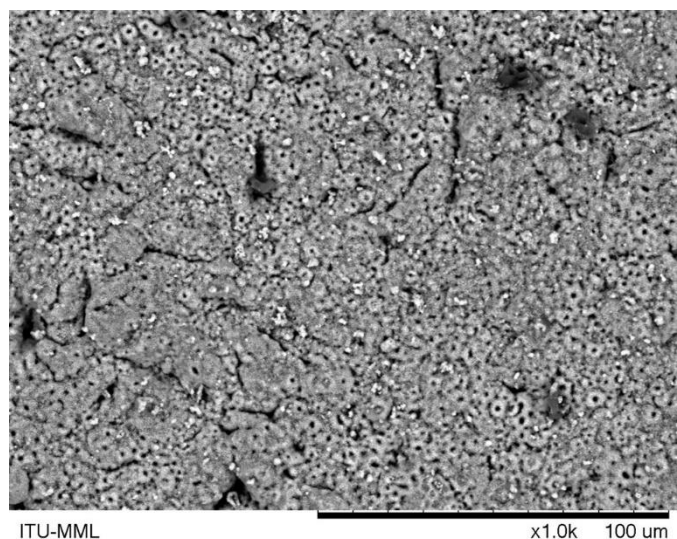


Figure 6.63 : F600-O disc SEM image.

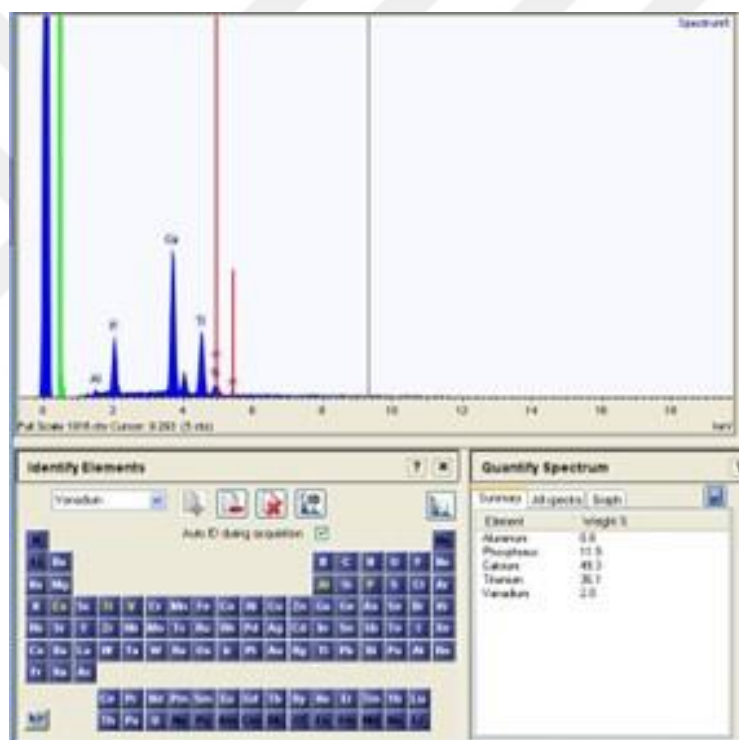


Figure 6.64 : EDS analysis of F600-O disc.

6.7 FTIR Analysis Results

In order to determine the functional groups and chemical bonds in the chemical structure of the under-studied samples, FTIR analysis was conducted and the obtained is shown in Figure 6.65 and 6.66.

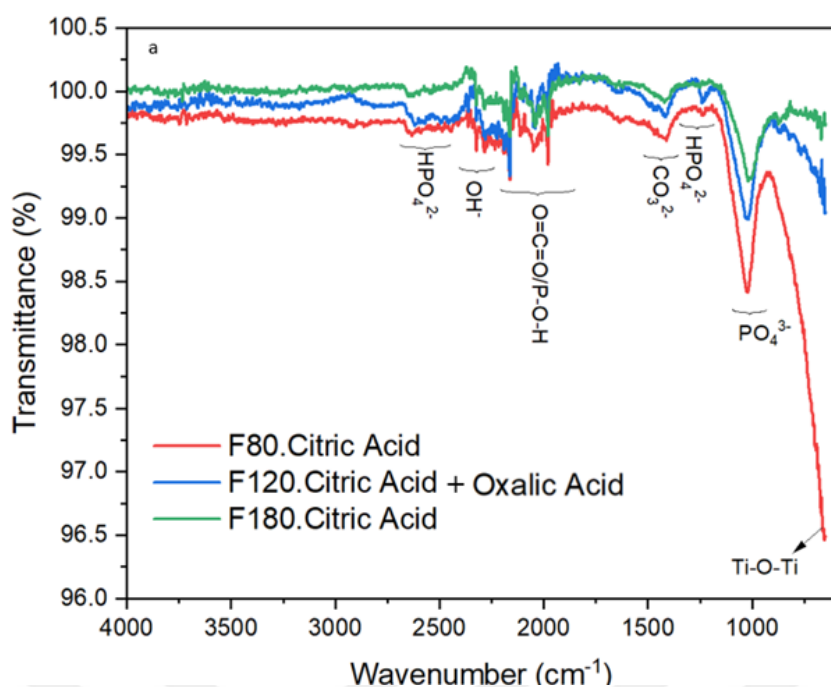


Figure 6.65 : Ftir analysis of SLA with citric acid etched and MAO treated sample after SBF.

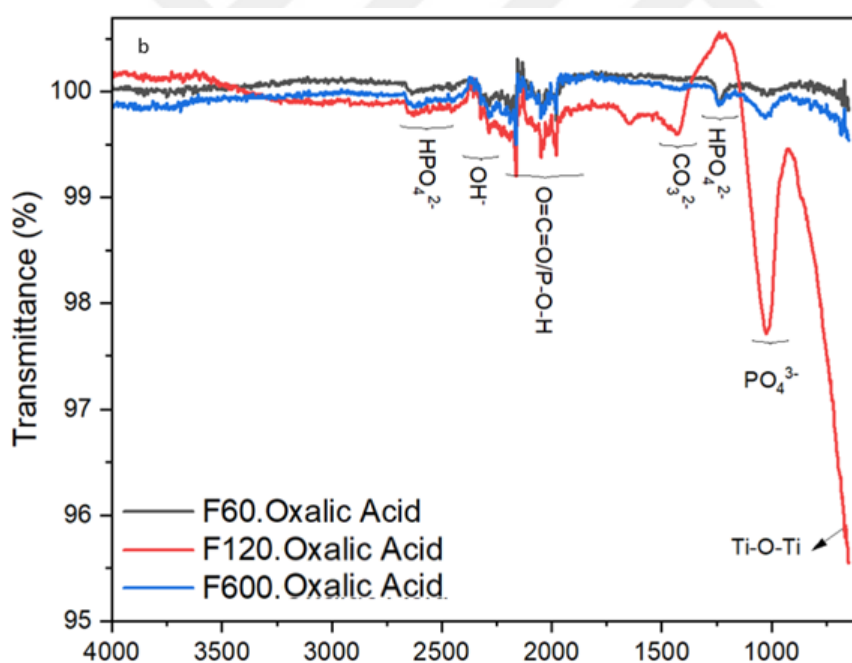


Figure 6.66 : Ftir Analysis of SLA with oxalic acid etched and MAO treated sample after SBF.

Table 6.2 shows the peak positions and related chemical bonds obtained from the FTIR spectra.

Table 6.2 : The peak positions and related chemical bonds obtained from FTIR spectra (ν : Stretching vibration, δ : Bending vibration).

Sample	ν (HPO_4^{2-})	δ (OH^-)	ν ($\text{O}=\text{C}=\text{O}/\text{P}-\text{O}-\text{H}$)	ν (CO_3^{2-})	δ (HPO_4^{2-})	ν (PO_4^{3-})	ν ($\text{Ti}-\text{O}-\text{Ti}$)
F80.Citric acid	2633 cm^{-1}	2283 cm^{-1}	2162 cm^{-1}	1412 cm^{-1}	1239 cm^{-1}	1013 cm^{-1}	654 cm^{-1}
F120.Citric + Oxalic acid	2619 cm^{-1}	2280 cm^{-1}	2163 cm^{-1}	1415 cm^{-1}	1241 cm^{-1}	1018 cm^{-1}	650 cm^{-1}
F180.Citric acid	2651 cm^{-1}	2287 cm^{-1}	2163 cm^{-1}	1421 cm^{-1}	1251 cm^{-1}	1019 cm^{-1}	670 cm^{-1}
F60.Oxalic acid	2633 cm^{-1}	2283 cm^{-1}	2167 cm^{-1}	1421 cm^{-1}	1239 cm^{-1}	1017 cm^{-1}	651 cm^{-1}
F120.Oxalic acid	2625 cm^{-1}	2282 cm^{-1}	2162 cm^{-1}	1411 cm^{-1}	1223 cm^{-1}	1013 cm^{-1}	650 cm^{-1}
F600.Oxalic acid	2625 cm^{-1}	2287 cm^{-1}	2162 cm^{-1}	1418 cm^{-1}	1241 cm^{-1}	1031 cm^{-1}	683 cm^{-1}

In these spectra, the band at about 2633 cm^{-1} was seen due to asymmetric and symmetric stretching vibrations of HPO_4^{2-} [232]. the characteristic peak around 2283 cm^{-1} is assigned to the OH^- stretching vibration [232], and the absorption band in the wavenumber range of 1900-2250 cm^{-1} is related to the stretching vibration of $\text{O}=\text{C}=\text{O}$ due to the CO_2 in the air [233,234] and the stretching vibration of $\text{P}-\text{O}-\text{H}$ [235]. In addition, the peaks located at 1421 cm^{-1} , 1239 cm^{-1} and 1017 cm^{-1} are attributed to vibration of $\text{C}-\text{O}$ in CO_3^{2-} [236,237], $\text{P}=\text{O}$ in HPO_4^{2-} [238], and $\text{P}-\text{O}$ in PO_4^{3-} in the chemical composition of hydroxyapatite [238, 239], respectively. The absorption band at the wavenumbers lower than 700 cm^{-1} is attributed to stretching vibration of metal-oxygen bonds ($\text{Ti}-\text{O}-\text{Ti}$) of the substrate [240]. Therefore, the FTIR analysis demonstrates the presence of hydroxyapatite on the $\text{Ti}_6\text{Al}_4\text{V}$ substrate.

From Figure 6.65 , observations indicated that by etching of the samples by citric acid, the intensity of the peak at around 1013 cm^{-1} and the peak at 1412 cm^{-1} increased and the peak positions shifted to higher wavenumbers (Table 6.2) as the alumina particle size decreases. The peak intensity at wavenumbers below 700 cm^{-1} in the sample sandblasted with F-80 alumina was significantly higher than other samples. In contrast, for other samples, this intensity decreased, and the peak positions shifted to higher wavenumbers (Table 6.2) with decreasing alumina particle size. Larger alumina particles create deeper and more pronounced roughness on the disk surface.

This higher surface roughness can lead to fewer active chemical sites and consequently a decrease in the intensity of peaks related to surface compounds. For instance, the

reduction in peak intensity around 1017 cm^{-1} and 1421 cm^{-1} can be attributed to the decrease in the number of active sites created during the sandblasting process. In other words, the size of the alumina particles can significantly impact the surface morphology and structure. Larger particles may alter the surface structure, increasing the sites where hydroxyapatite compounds can form. This can explain the observed increase in the intensities of the peaks in the range of 1017 cm^{-1} and 1421 cm^{-1} . In addition, in samples sandblasted with larger particles, there is deeper holes available for chemical reactions and the formation of hydroxyapatite layers. Consequently, the intensity of the peaks related to Ti-O-Ti bonds at wavenumbers below 700 cm^{-1} is higher in samples treated with larger alumina particles compared to others. This indicates better surface activation and higher reaction rates in samples treated with larger particles. For the samples etched with oxalic acid, among those sandblasted with alumina particles of different diameters, the most intense peaks corresponding to phosphate, carbonate, and titanium oxide groups were observed in the sample sandblasted with F-120 particles. Additionally, the positions of these peaks shifted to lower wavenumbers in the presence of this particle size compared to other samples. The F-120 alumina particles likely create an optimal surface roughness that enhances physical and chemical activation of the Ti6Al4V alloy surface. This increased roughness results in more nucleation sites for the formation of hydroxyapatite groups, leading to more intense FTIR peaks. In fact, larger particles could cause excessive surface damage, leading to fewer active sites, while smaller particles might not impart enough energy to sufficiently modify the surface. The intermediate size allows for effective surface roughening without overly damaging the substrate, creating a favorable environment for subsequent chemical reactions. The shift of peak positions to lower wavenumbers in samples treated with F-120 particles suggests changes in the chemical environment of the surface functional groups. This shift may indicate stronger or more stable chemical bonds, possibly due to an ideal combination of surface roughness and chemical activation. These changes in the local electronic environment lead to shifts in the vibrational frequencies of the functional groups, observed as peak shifts in the FTIR spectrum. Therefore, the specific roughness achieved with F-80 and F-120 particles may be ideal for enhancing surface energy and interaction with etching agents and subsequent treatments.

6.8 XRD Analysis

After the MAO treatment and immersion in SBF, XRD analysis of 6 samples was performed. The XRD analysis of the samples are given in Figure 6.67.

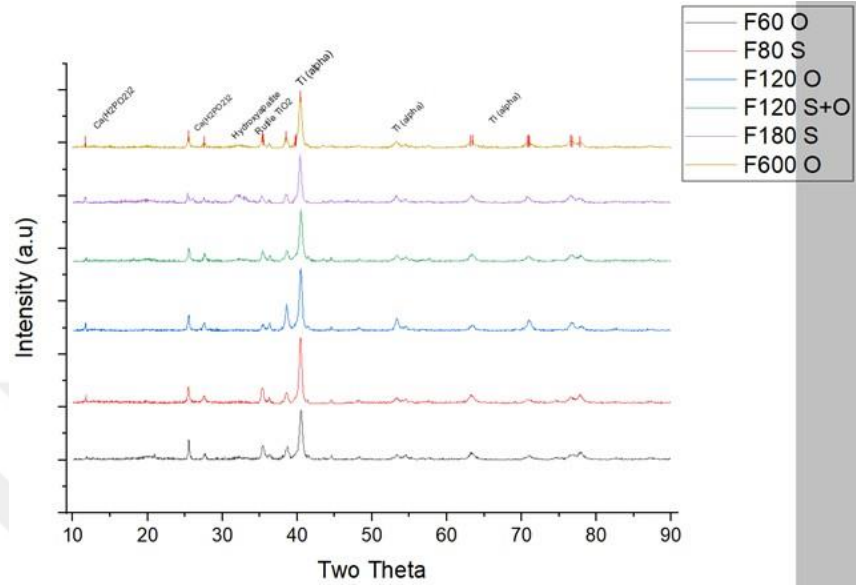


Figure 6.67 : XRD analysis result.

XRD (X-ray Diffraction) analysis is a method used to determine the crystal structure, phase composition and crystal dimensions of the material. The XRD analysis performed in this section was used to evaluate whether the oxide layers expected to form after the MAO process were formed and the apatite structures expected to form after SBF.

In all the XRD analysis obtained, the main phase of the Ti-6Al-4V alloy, α -Ti, is first observed. Since it is the main material of the sample's substrate, it is a phase expected to be dense and is observed densely. These peaks can be detected thanks to the use of X-rays and the porous structure of the oxide layer formed with MAO.[241] Another detected phase is the Rutile TiO₂ phase. The peaks belonging to the Rutile TiO₂ (01-076-1938) phase are seen at approximately 27°, 36° and 54°. When similar studies are examined, the detection of this phase confirms that the MAO process has been successfully completed and an oxide layer in the form of TiO₂ has formed on the surface. [242] Another phase, the Hydroxyapatite phase (01-074-0566), has been identified and is especially seen in peaks around 26° and 32°. It is the bioactive phase formed on the surface after being deposited in the SBF solution.

The peak intensity indicates the amount of apatite formation. These peaks confirm the formation of hydroxyapatite on the surface after being deposited in the SBF solution. In other words, it shows that the sample is biocompatible and has osteointegration potential. [243,244] The phases contained in each sample and their comparisons according to XRD analysis are given in table below;

Table 6.3 : Phases their comparisons according to XRD analysis.

Sample	Grain Size and Acid Type	Rutile TiO ₂	Hydroxyapatite	Apatite Preliminary Phase (Ca(H ₂ PO ₄) ₂)
F60 + O	212-300 µm, oxalic acid	✓	✓	✓
F80 + C	150-212 µm, citric acid	✓	✓ (Most Intense)	✓
F120 + O	90-120 µm, oxalic acid	✓	✓ (Intense)	✓
F120 C+O	90-120 µm, citric + oxalic acid	✓	✓ (Intense)	✓
F180 C	63-90 µm, citric acid	✓	✓	✓
F600 O	3-19 µm, oxalic acid	✓	✓ (Lowest Density)	✓

In the obtained data, F80 S sample showed the most intense hydroxyapatite peak, while F120 S+O showed the highest peaks immediately after F80 sample. The lowest apatite presence was detected in F600 O sample. It was thought that excessively small sand grain size had a negative effect on apatite formation, medium sand sizes contributed to more apatite formation and therefore could be the best surface for supporting bone cell adhesion and formation.

Rutile TiO₂ peak appeared stronger in F80 and F120 samples. However, the presence of Ca(H₂PO₄)₂ phase seen in all samples indicates that apatite formation is not complete and longer SBF immersions are required. Thus, it is thought that apatite crystallization can be increased.

7. DISCUSSION

The analysis images and analysis results obtained were compared with the literature. In the first stage, sandblasting results were taken as basis. When the obtained SEM images were compared with the literature, results with similar appearances and similar roughness values were encountered in the literature. As a result of the comparison, the field depths and homogeneous rough surface images on the surfaces of other SLA (sandblasting-acid etching) surface modified implant samples and disks confirm that sandblasting and acid etching processes were performed. Surface roughness measurements were taken with the profilometry device and it was seen that it was close to the surface roughness values accepted as ideal in the range of 1-2 μm . Surface roughness measurements of the samples are shown in Table 7.1 and Table 7.2.

Table 7.1 : Surface roughness measurements of implants.

Sample	Ra1	Rz1	Ra2	Rz2
90-125- μm alumina +oxalic acid	1.58	7.07	1.47	5.99
212-300 μm alumina + citric acid	1.34	6.89	1.49	7.77
63-90 μm alumina + citric acid	1.44	4.72	1.25	6.82
90-125- μm alumina +citric acid	1.58	6.56	1.04	4.8
90-125- μm alumina +oxalic acid	0.81	3.71	1.25	5.76
150-212 μm alumina +citric acid	1.15	6.47	1.01	4.82
150-212 μm alumina +oxalic acid	0.86	4.97	1.11	5.11
212-300 μm alumina + oxalic acid	1.17	6.48	1.27	5.82
3-19 μm alumina + oxalic acid	0.93	4.28	0.86	3.48
63-90 μm alumina + oxalic acid	0.99	4.88	0.51	3.08
150-212 μm alumina + citric + oxalic acid	0.93	5.45	0.79	4.22
3-19 μm alumina + citric+ oxalic acid	0.73	3.75	0.69	3.81
212-300 μm alumina + citric+ oxalic acid	1.2	6.5	0.96	4.83
63-90 μm alumina + citric+ oxalic acid	0.88	3.47	1.07	4.04
90-125- μm alumina + citric + oxalic acid	0.76	4.66	0.85	4.9

Table 7.2 : Surface roughness measurements of disc samples.

Sample	Ra
3-19 μm alumina + citric acid	0,26
63-90 μm alumina + oxalic acid	0,70
90-125- μm alumina +citric acid	1,05
212-300 μm alumina + citric acid	1,16
212-300 μm alumina + citric+ oxalic acid	2,02
63-90 μm alumina + citric+ oxalic acid	0,93
150-212 μm alumina +citric acid	0,91
150-212 μm alumina +oxalic acid	1,28
150-212 μm alumina + citric + oxalic acid	1,64
3-19 μm alumina + citric + oxalic acid	0,27
212-300 μm alumina + oxalic acid	1,18

In the next stage, SBF (Simulated Body Fluid) was prepared. The prepared disc samples were immersed in the liquid and waited for 14 days. At the end of the period, SEM-EDS images were taken. When the obtained images were examined, it was seen that apatite structures were not formed. Afterwards, Micro Arc Oxidation processes were carried out using a 30 kW capacity bipolar power source. In this setup, a stainless steel container containing electrolyte was used as the cathode and the sample was used as the anode. This surface, which formed an oxide layer, was immersed in SBF again and waited. The samples removed from SBF were washed with deionized water, dried and then re-analyzed. When the analysis results were compared with the literature, cumulative and aggregative apatite structures were observed. Afterwards, XRD and FTIR analyses were applied. While the α -Ti peak was dominant in the findings obtained from the XRD results, it was observed that this peak was accompanied by rutile TiO_2 , Hydroxylapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) and ($\text{Ca}(\text{H}_2\text{PO}_2)_2$) peaks. The TiO_2 peak seen in all samples shows that the oxide layer was successfully obtained. The Hydroxyapatite peak obtained in all results emphasizes that apatite structures were successfully formed and a surface with bioactive properties was created. Then in order to determine the functional groups and chemical bonds in the chemical structure of the under-studied samples, FTIR analysis was conducted. From FTIR analysis observations indicated that by etching of the samples by citric acid, the intensity of the

peak at around 1013 cm^{-1} (Phosphate) and the peak at 1412 cm^{-1} (Carbonate) increased and the peak positions shifted to higher wavenumbers as the alumina particle size decreases. The peak intensity at wavenumbers below 700 cm^{-1} in the sample sandblasted with F-80 alumina was significantly higher than other samples. In contrast, for other samples, this intensity decreased, and the peak positions shifted to higher wavenumbers with decreasing alumina particle size. Larger alumina particles create deeper and more pronounced roughness on the disk surface. This higher surface roughness can lead to fewer active chemical sites and consequently a decrease in the intensity of peaks related to surface compounds. For instance, the reduction in peak intensity around 1017 cm^{-1} and 1421 cm^{-1} can be attributed to the decrease in the number of active sites created during the sandblasting process. For the samples etched with oxalic acid, among those sandblasted with alumina particles of different diameters, the most intense peaks corresponding to phosphate, carbonate, and titanium oxide groups were observed in the sample sandblasted with F-120 particles.

Additionally, the positions of these peaks shifted to lower wavenumbers in the presence of this particle size compared to other samples. F-80 and F-120 alumina particles likely create an optimal surface roughness that enhances physical and chemical activation of the Ti6Al4V alloy surface. This increased optimum roughness results in more nucleation sites for the formation of hydroxyapatite groups, leading to more intense FTIR peaks.

7.1 Detection of Hydroxyapatite Formation in Samples Using XPS Analysis

In Figure 7.1, the hydroxyapatite formation of the samples was determined with a) F600 O (with 3-19 micrometer size, used for the acid etching in oxalic acid), b) F120 C+O (90-12 micrometer size, used for the acid etching in citric acid + oxalic acid), c) F80 C (150-212 micrometer size, used for the acid etching in citric acid), and d) F120 C (90-125 micrometer size, used for the acid etching in citric acid), respectively. In the XPS analysis, Ca 2p peak intensities were determined as F600 O>F120 C+O>F80 C >F120 C, respectively. Calcium (Ca) peak was determined in the hydroxyapatite (HA) analysis. Because one of the basic components of HA is Ca. etc.) presence is indicated. Ca 2p binding energies (BE) and chemical shifts were investigated. The Ca 2p_{3/2} peak for hydroxyapatite was detected around 347.1–347.5 eV, where it should be. The differences in Ca 2p peak intensities in the XPS analysis are due to various factors

such as the type of acid used, particle size and surface morphology. The reasons for these differences are:

- 1) Effect of the type of acid, while the F600 O sample was etched with only oxalic acid, the other samples were processed with a combination of citric acid and oxalic acid and only citric acid. Oxalic acid affected the Ca content on the surface as a result of calcium oxalate formation. A suitable environment was provided for hydroxyapatite formation (providing the appropriate pH value and formation of sufficient phosphate ions). Thus, calcium oxalate directly increased the formation of HA without accumulating Ca on the surface. The fact that F600 O showed the highest Ca 2p peak indicated that oxalic acid contributed more to the formation of hydroxyapatite.
- 2) Effect of particle size, Small particles (F120 C+O and F120 C) have a larger specific surface area, which allows more surface reactions. This also caused Ca to dissolve and move away and the Ca content on the surface to decrease. F600 O (3-19 μm) has the
- 3) smallest particle size and shows the highest Ca 2p peak intensity, which allowed more Ca content to remain on the surface.
- 4) The factors affecting the formation of hydroxyapatite, the type of acid used and the etching process, affected the binding of Ca ions on the surface to the HA structure or their dissolution and removal. F600 O has the highest Ca 2p intensity, which strengthens the bond between the bone and the implant and increases biocompatibility. Thus, it ensured that hydroxyapatite was formed the most in this sample.
- 5) Chemical shift and binding energies, XPS analysis, Ca 2p_{3/2} peak in the range of 347.1–347.5 eV confirmed the successful formation of hydroxyapatite.
- 6) F120 C has the lowest Ca 2p peak, indicating that this surface treatment contributes less to HA formation.

It was determined that hydroxyapatite formation varies depending on the type of acid used, particle size and surface treatment parameters. This information helped to select the most appropriate surface modification method for dental implants.

Fig. 7.2 P 2p peak intensities of a) F120 C, b) F120 C+O, c) F80 C and d) F600 O samples are indicated. According to XPS analysis, the samples where P 2p is most intense are detected as F120C > F120C+ > F80C > F600 O, from highest to lowest intensity of P 2p peaks. The formation of phosphorus (P 2p) peak was also examined to confirm the formation of hydroxyapatite, and the binding energy of the P 2p peak was detected at the place where it should be around ~133 eV. When the intensity of P 2p peaks is compared, the reasons why the samples where P 2p is most intense (from highest to lowest intensity of P 2p peaks) are detected as F120C > F120C+O > F80C > F600 O are:

- 1) Since the P signal is stronger in F120C and F120C+O, it is thought that there is a phase containing more phosphorus (hydroxyapatite) on the surface in these samples.
- 2) It was determined that the F600 O sample was the sample with the weakest P signal. The reason for the less hydroxyapatite in the F600 O sample was that it was thermally treated at 50 C and chemically treated in oxalic acid, which caused phosphorus to diffuse more from the surface and cause more dissolution.
- 3) Since the P 2p signal was detected as strong, there is hydroxyapatite formation on the surface. Because, when the binding energies are examined, the P 2p peak for hydroxyapatite is generally around ~133-134 eV. There is no shift outside this range. Therefore, it was thought that different phosphate phases (such as calcium phosphate, tricalcium phosphate) were not present on the surface. Thus, it was more clearly evaluated that hydroxyapatite was dominant on the surface.

In general, when the P 2p and Ca 2p results were evaluated together in XPS analyses, the hydroxyapatite formation occurred the most in the F120 C sample. In the F600 O sample, the P2p signal was weakened, indicating that the amount of HA decreased due to oxalic acid. The F120C+O sample may be the next best sample in terms of hydroxyapatite, but different changes occurred on the surface due to the effect of oxygen.

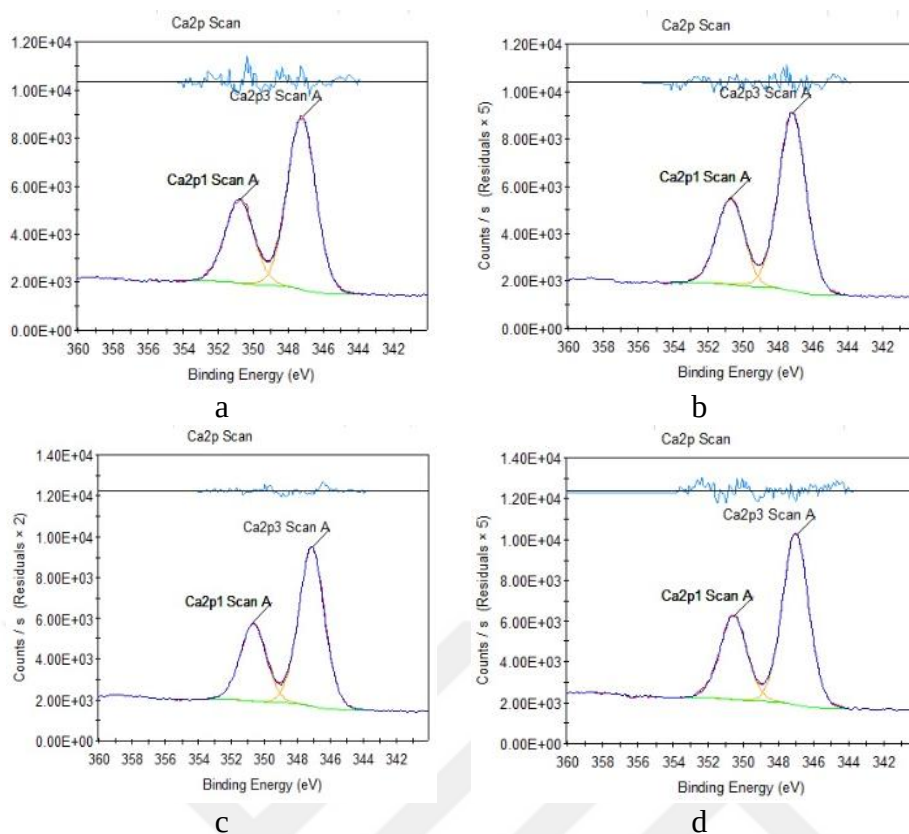


Figure 7.1 : Ca 2p peak intensities of a) F600 O, b) F120 C+O, c) F80 C and d) F120 C samples.

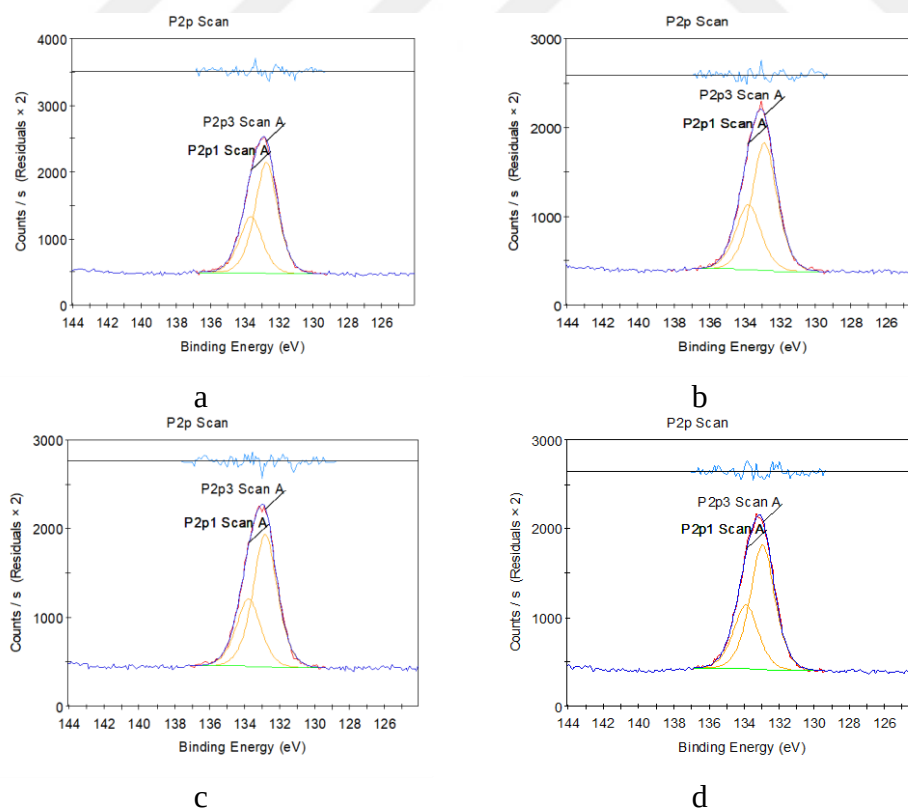


Figure 7.2 : P 2p peak intensities of a) F120 C, b) F120 C+O, c) F80 C and d) F600 O samples.

8. CONCLUSIONS

This thesis aims to determine how implant success can be achieved by evaluating the factors that may cause implant failure. In the first stage, SEM images of implants that could not provide osseointegration were taken. In the SEM images, structures such as mechanical deformation, micro-cracks, and heterogeneous distribution in the surface coating were observed. Then, radiological images were taken to determine the presence of fractures in their internal structures. No fracture structures were observed in the radiological images taken using X-rays. One of the most important factors in implant success is surface roughness. Therefore, by using sands of different sizes, the effect of sands on implant success was investigated, and it was aimed to increase the bioactivity of the surface by performing micro-arc oxidation. There is a correlation between in vivo bone growth and in vitro apatite formation ability. For this reason, SLA surface modification was performed using citric acid and oxalic acid on the disc surfaces roughened using 3-19 μm , 63-90 μm , 90-125 μm , 150-212 μm and 212-300 μm aluminum oxide sands. In order to compare the bioactivities of these samples before and after, SBF was prepared and immersed in this liquid. No apatite-like structures were observed in the SEM images obtained at the end of the 14-day waiting period. Then, these samples were prepared for MAO process. MAO process was carried out using 20.5 g/L Calcium Acetate ($\text{C}_4\text{H}_6\text{CaO}_4$) and 14.4 g/L Monosodium phosphate (NaH_2PO_4) in a total of 2.5 liters of solution. The process, in which 300 V positive voltage and 60 V negative voltage were applied, lasted for 3 minutes. Then the samples were immersed in SBF again. In the obtained SEM images, apatite structures were clearly seen and confirmed by comparing with the literature. In the SEM-EDS analysis, it is seen with the presence of Ca and P. When this result is compared with the disks applied only SLA, it seems that the MAO treatment positively contributes to the bioactivity of the surface. The data obtained from FTIR and XRD confirms the formation of titanium oxide layer and apatite structures.

In the data obtained here, it was found that particles larger than the optimum particle size could cause excessive surface damage, leading to fewer active sites, while smaller

particles may not provide enough energy to sufficiently modify the surface. The intermediate size allows for effective surface roughening without overly damaging the substrate, creating a favorable environment for subsequent chemical reactions. The shift of peak positions to lower wavenumbers in samples treated with F-80 and F-120 particles suggests changes in the chemical environment of the surface functional groups. This shift may indicate stronger or more stable chemical bonds, possibly due to an ideal combination of surface roughness and chemical activation. These changes in the local electronic environment lead to shifts in the vibrational frequencies of the functional groups, The intermediate size allows for effective surface roughening without overly damaging the substrate, creating a favorable environment for subsequent chemical reactions. The shift of peak positions to lower wavenumbers in samples treated with F-80 and F-120 particles suggests changes in the chemical environment of the surface functional groups. This shift may indicate stronger or more stable chemical bonds, possibly due to an ideal combination of surface roughness and chemical activation. These changes in the local electronic environment lead to shifts in the vibrational frequencies of the functional groups, observed as peak shifts in the FTIR spectrum. Therefore, the specific roughness achieved with F-80 and F-120 particles may be ideal for enhancing surface energy and interaction with etching agents and subsequent treatments observed as peak shifts in the FTIR spectrum. Therefore, the specific roughness achieved with F-80 and F-120 particles may be ideal for enhancing surface energy and interaction with etching agents and subsequent treatments.

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