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STUDIES AND RESEARCH REGARDING THEMODIFICATION OF THE SURFACE OF DENTAL IMPLANTS USING THE ABRASION PROCESS

STUDII SI CERCETĂRI PRIVIND MODIFICAREASUPRAFEȚEI IMPLANTURILOR DENTARE UTILIZÂND PROCESUL DE ABRAZIUNE

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Table of Contents

PART I

CHAPTER 1. GENERAL CONSIDERATIONS REGARDING DENTAL IMPLANTS AND BIOMATERIALS USED

Dental implants represent one of the most significant innovations in dentistry, with a long history and substantial advancements in both the materials used and the insertion techniques. The need to replace tooth loss due to disease or trauma has existed since ancient times, with early restoration attempts involving primitive materials such as wood, shells, stone, or carved ivory. Over the centuries, scientific progress has led to the use of metals, ceramics, and synthetic materials, significantly increasing the success rate of implants.

Dental implants are classified based on the surgical method used for insertion. Subperiosteal implants are placed on the alveolar bone, beneath the periosteum, and are made from custom metal frameworks. Transosseous implants pass through the mandibular bone and are used only in specific cases. The most commonly used category is endosseous implants, which are inserted directly into the jawbone and come in various shapes, including cylindrical and threaded designs. Among these, root-form implants, whether threaded or self-tapping, are the most widely used due to their long-term stability and effective osseointegration.

The materials used for implant fabrication have evolved significantly, influenced by research on their biocompatibility and durability. Biomaterials used in implantology can be classified into three generations. The first generation consisted of passive materials, such as pure titanium and ceramics. The second generation introduced bioactive materials that interact with the biological environment, such as hydroxyapatite. Currently, the third generation is dominated by smart biomaterials, which can respond to biological stimuli and contribute to tissue regeneration.

Among the materials used in implantology, titanium and titanium alloys are the most common due to their high strength and excellent osseointegration capability. Titanium quickly forms a protective oxide layer that prevents corrosion and ensures high biological compatibility. Another important material is zirconia, valued for its superior aesthetics, corrosion resistance, and excellent biocompatibility. Titanium-zirconium alloys combine the advantages of both materials, providing enhanced mechanical strength and improved osseointegration.

The design of dental implants significantly influences their success. The shape, thread geometry, and implant surface play a crucial role in stability. For example, threaded implants maximize bone-to-implant contact, enhance primary stability, and improve the distribution of applied forces. Implant diameter and length are also key factors, affecting mechanical strength and stress distribution on the bone. A larger diameter helps reduce tension at the crestal bone level, while implant length can impact primary stability. Studies indicate that most stress is concentrated in the crestal third of the implant, and variations in implant length do not significantly affect stress distribution.

The efficiency of dental implants is influenced by several factors, including the biocompatibility of the material, implant-abutment connection, surgical technique, and patient conditions. A key factor is the distribution of forces applied to the implant. Axial forces are the most favorable, as they generate compressive stress, which the bone can better tolerate. *(Figure 1.12)* On the other hand, bending moments caused by transverse forces can lead to the formation of microcracks and marginal bone loss. *(Figure 1.13)*

Figure 1.12. Axial load distribution. The morphology of the threads can influence the nature of the compressive force created in the bone surrounding a machined implant. [1]

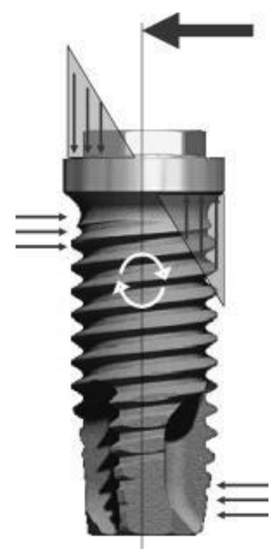


Figure 1.13. Transverse forces. Lateral forces challenge both the fixation body and the interface with the crestal bone. [1]

CHAPTER 2. AIR ABRASIVE DENTAL SYSTEMS AND CERAMIC POWDERS USED IN DENTISTRY

Air abrasive dental systems and ceramic powders represent a significant innovation in modern dentistry, enabling minimally invasive treatments. These technologies are applied in a variety of dental procedures, including surface cleaning, removal of bacterial plaque and stains, cavity preparation, and polishing of restorative materials. Unlike traditional methods that involve rotary burs, air abrasive dental systems provide a high level of precision, significantly reduce patient discomfort, and prevent overheating of dental tissues, which are essential factors for an efficient and comfortable treatment.

Figure 2.1. Air abrasion in dentistry [2]

The operating principle of these systems is based on the propulsion of fine abrasive particles, such as aluminum oxide or sodium bicarbonate, towards the dental surface using a controlled flow of compressed air or an air-water mixture. These particles strike the dental structure with enough energy to remove enamel, dentin, or restorative materials without causing significant damage to the surrounding tissues. The air pressure used in these systems generally ranges from 40 to 120 psi, and the abrasive particles are evenly distributed to ensure an effective treatment. The system consists of a reservoir for abrasive particles connected to a compressed air source, a specialized handpiece that allows precise direction of the abrasive flow, and a pressure and distribution control mechanism for adjusting the intensity of the process.

The main applications of air abrasive dental systems include dental cleaning and prophylaxis, where they effectively remove bacterial plaque and stains from the enamel surface. They are also used in cavity preparation, allowing for the minimally invasive removal of carious structures without the need for rotary burs, thereby preserving more healthy tooth tissue.

Compared to conventional treatment methods, air abrasion technology offers numerous advantages. One of the key benefits is its minimally invasive nature, which allows for the preservation of a greater amount of healthy dental tissue. Additionally, it eliminates vibrations and excessive heat, providing increased comfort for the patient. The procedure also ensures high precision, as the abrasive particles can be directed in a controlled manner, and it is highly effective in decontaminating dental implant surfaces. However, there are certain limitations to consider. The technique requires specialized equipment, it is ineffective in removing hard restorative materials, and there is a risk of inhaling abrasive particles, necessitating additional protective measures for both the dentist and the patient.

The air abrasion system, traditionally used for cleaning dental surfaces, has been investigated as an alternative method for applying bioceramic coatings. Compared to conventional techniques, this method offers superior control over the thickness of the applied layer, surface roughness, and uniformity. By adjusting the abrasion parameters, implant surface characteristics can be modified to optimize osseointegration.

Another innovative application of the air abrasion system is its use in coating dental implants with bioceramic materials, such as hydroxyapatite and calcium phosphate. These coatings play a crucial role in enhancing osseointegration and reducing the risk of implant rejection. They offer multiple advantages, including improved bioactivity, enhanced osteoblast attachment, and precise control over the thickness of the applied layer. Unlike traditional methods such as plasma spraying, air abrasion prevents thermal degradation of the material, preserving its structural integrity and biological effectiveness.

Compared to other bioceramic coating application methods, air abrasion offers several advantages. It enhances bioactivity by promoting osteoblast adhesion and bone integration. Unlike techniques that involve high temperatures, such as plasma spraying, air abrasion does not generate excessive heat, thereby preventing material degradation. Additionally, it allows for precise control over coating thickness, optimizing the surface properties of the implant. Moreover, this method is both efficient and cost-effective, requiring less complex equipment than advanced techniques, making it a practical and revolutionary solution for improving dental and orthopedic implants.

PART II

CHAPTER 3. OBJECTIVES OF THE THESIS AND RESEARCH METHODOLOGY FOR THE CHARACTERIZATION OF TITANIUM DENTAL IMPLANT SURFACES

Dental implants are a modern and effective solution for replacing missing teeth, but their success depends on multiple factors, including the biocompatibility of the material used and the surface properties of the implant. Over time, the materials used in implantology have evolved, with titanium and its alloys, as well as zirconia, being the most widely used. These materials differ in their mechanical, physical, and chemical properties, which directly impact the rate of osseointegration and the overall biofunctionality of the implant.

The surface of the dental implant is essential for facilitating bone-to-implant contact and ensuring successful osseointegration. In recent years, modifications to dental implant surfaces have been extensively studied to optimize this process. Various surface treatment techniques have been developed to enhance the interaction between the implant and the bone. These modifications can be physical, chemical, or a combination of both, each aimed at improving the implant's performance and integration.

Chemical and physical properties of the implant surface

The chemical composition of an implant's surface is influenced by the nature of the material from which it is made. A relevant example is the Ti6Al4V alloy, whose corrosion resistance is attributed to the formation of a thin titanium oxide layer. This passive layer protects the implant from degradation and can mimic the ceramic structure of bone, thereby facilitating osseointegration.

The surface topography of an implant directly influences the biological response of the bone. Studies have shown that rough-surfaced implants achieve better osseointegration compared to smooth-surfaced implants. This is due to the structural complexity of rough surfaces, which promotes bone tissue formation and anchorage. To mimic the structure of human bone, implants undergo surface modification processes at a microscopic scale.

Another important factor is the surface free energy of the implant, which influences its interaction with biological tissues. Hydrophilic surfaces have been associated with faster osseointegration and better bone cell proliferation compared to hydrophobic surfaces.

Methods for surface modification of implants

To enhance the properties of dental implants, their surface can be treated using various methods:

- Sandblasting with titanium or aluminum oxide particles to create a rough surface that promotes bone anchorage.

- Acid etching, which improves the adhesion of bone cells to the implant surface.

- Hydroxyapatite (HA) coating, a bioceramic material similar to bone structure, which stimulates bone tissue formation.

During the healing process, bone cells gradually attach to the implant surface, and this process is significantly accelerated in the case of treated surfaces. On smooth surfaces, the biological interaction between bone and implant occurs at a slower rate, making implant integration more challenging.

Another technique used for implant surface treatment is the deposition of calcium phosphate-based bioceramics. The release of calcium and phosphorus ions in the implant area promotes the formation of a biological apatite layer, which acts as a matrix for bone growth.

Research methodology

To analyze the properties of dental implant surfaces, several investigation methods were used:

- Scanning Electron Microscopy (SEM) to examine surface morphology.

- Energy Dispersive X-ray Spectroscopy (EDS) to determine the chemical composition.

- X-ray Diffraction (XRD) to characterize the crystalline structure of the materials used.

- Fourier Transform Infrared Spectroscopy (FT-IR) to identify chemical functional groups present on the surface.

The aim of the research was to analyze the effect of surface modification on titanium alloy implants (Ti6Al4V) by applying a hydroxyapatite coating. The study focused on evaluating the morphological, structural, and chemical characteristics of the treated implant surfaces, as well as their impact on osseointegration.

3.1. Materials used, experimental sample preparation, and evaluation methods

In this study, the analyzed dental implants were made of Ti6Al4V titanium alloy, a material known for its biocompatibility and superior strength. The implants, supplied by Biotec Dental Implants System, had standardized dimensions of 3.0 mm in diameter and 11.5 mm in length. These dimensions make them suitable for patients with narrow dental spaces or low bone density. The studied implants underwent advanced surface treatments to enhance osseointegration, the essential process through which the implant becomes an integral part of the jawbone.

An important factor in the integration of implants into the bone structure is their surface texture. Studies show that implants with treated surfaces exhibit faster osseointegration and greater stability compared to those with smooth surfaces. To enhance these characteristics, the implants used in this research were treated using two methods:

- SLA (Sandblasting + Acid Etching) – a technique that creates micro-cavities on the implant surface through a combination of mechanical sandblasting and chemical etching, promoting cell adhesion.

- RBM (Resorbable Blast Media) – a method that physically modifies the implant surface using biocompatible, resorbable particles, facilitating cohesion with bone tissue.

For this research, the surface of the implants was modified using the SLA method, which involved sandblasting with hard alumina particles, followed by a thorough cleaning process using acetone, ethanol, and distilled water. Afterward, the implants underwent a chemical treatment with a mixture of sulfuric acid (48%), hydrochloric acid (18%), and nitric acid (8%), and the process was completed by neutralizing the surface with a sodium bicarbonate solution. The goal of this procedure was to create a porous surface, which enhances the interaction between the implant and the bone, promoting better osseointegration. *(Figure 3.1)*

Use of hydroxyapatite for implant surface enhancement

To enhance osseointegration, hydroxyapatite was used, a mineral compound that mimics the structure of human bone. The hydroxyapatite powder used in this study was sourced from Plasma Biotal Limited UK and was applied to the implant surface using an air abrasion process. This treatment aimed to improve cell adhesion, stimulate bone regeneration, and create a biologically active layer that facilitates integration with surrounding tissues.

Hydroxyapatite is a bioactive material with the ability to interact with bone tissue, facilitating a direct bond between the implant and the bone. In its fine powder form, it was used to clean the implant surface without causing any structural damage. *(Figure 3.2).*

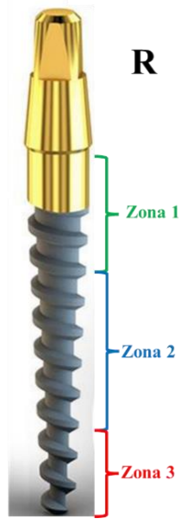


Figure 3.1. Appearance of the implants used in the study, highlighting the areas of interest investigated.

Figure 3.2. Appearance of the hydroxyapatite powder used in the experiment.

Preparation of experimental samples

The implant surface modification process was carried out at the National University of Science and Technology Politehnica Bucharest, using specialized equipment for depositing materials onto metallic substrates. The procedure involved exposing the implant to a controlled flow of hydroxyapatite particles, ensuring a uniform coating of its surface. Subsequently, the modified samples underwent detailed analyses to evaluate the distribution and adhesion of the hydroxyapatite layers.

Methods for implant surface analysis

To evaluate the impact of surface modifications on dental implants, several advanced analysis methods were used:

⇾ Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

This method was used to analyze the morphology of the implant surface, texture, chemical composition, and structural characteristics. SEM uses a beam of electrons to examine the implant surface at a microscopic scale, providing detailed information about its topography and structure.

⇾ Fourier Transform Infrared Spectroscopy (FT-IR)

This method allows for the identification of the chemical compounds present on the surface of the implant and is used to analyze the structural modifications resulting from the surface treatment.

⇾ X-ray Diffraction (XRD)

Used to determine the crystalline structure of the implant surface, this technique highlights the compositional changes produced as a result of the applied treatments.

⇾ Determination of Implant Surface Roughness

The surface roughness influences the degree of osseointegration and the stability of the implant. Roughness measurements were performed according to the international standard ISO 21920-1:2021, which classifies implant surfaces based on their roughness level.

⇾ Evaluation of Implant Surface Wettability

The wettability degree is determined by measuring the contact angle between a droplet of liquid and the implant surface. Hydrophilic surfaces, with a contact angle of less than 90°, promote osseointegration, while hydrophobic surfaces (contact angle >90°) can hinder this process.

3.2. Experimental results and discussions

The analysis of dental implant surfaces is a crucial aspect in improving osseointegration and the long-term success of implants. In this subsection, we studied the morphology and chemical composition of Ti6Al4V titanium alloy implants with an untreated surface, used as a reference sample (R). The study was conducted using advanced techniques such as Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS), as well as by analyzing the surface roughness and wettability.

Evaluation of the morphology and elemental composition of the reference sample surface (R)

To understand the structure of the dental implant surfaces, the samples were analyzed in three distinct areas. SEM images highlighted the threaded design of the implant and the microscopic differences resulting from the manufacturing processes. It was observed that the implant surface exhibits micro-cavities and uniform roughness, characteristics that are favorable for osseointegration. *(Figure 3.8; Figure 3.9; Figure 3.10; Figure 3.11)*

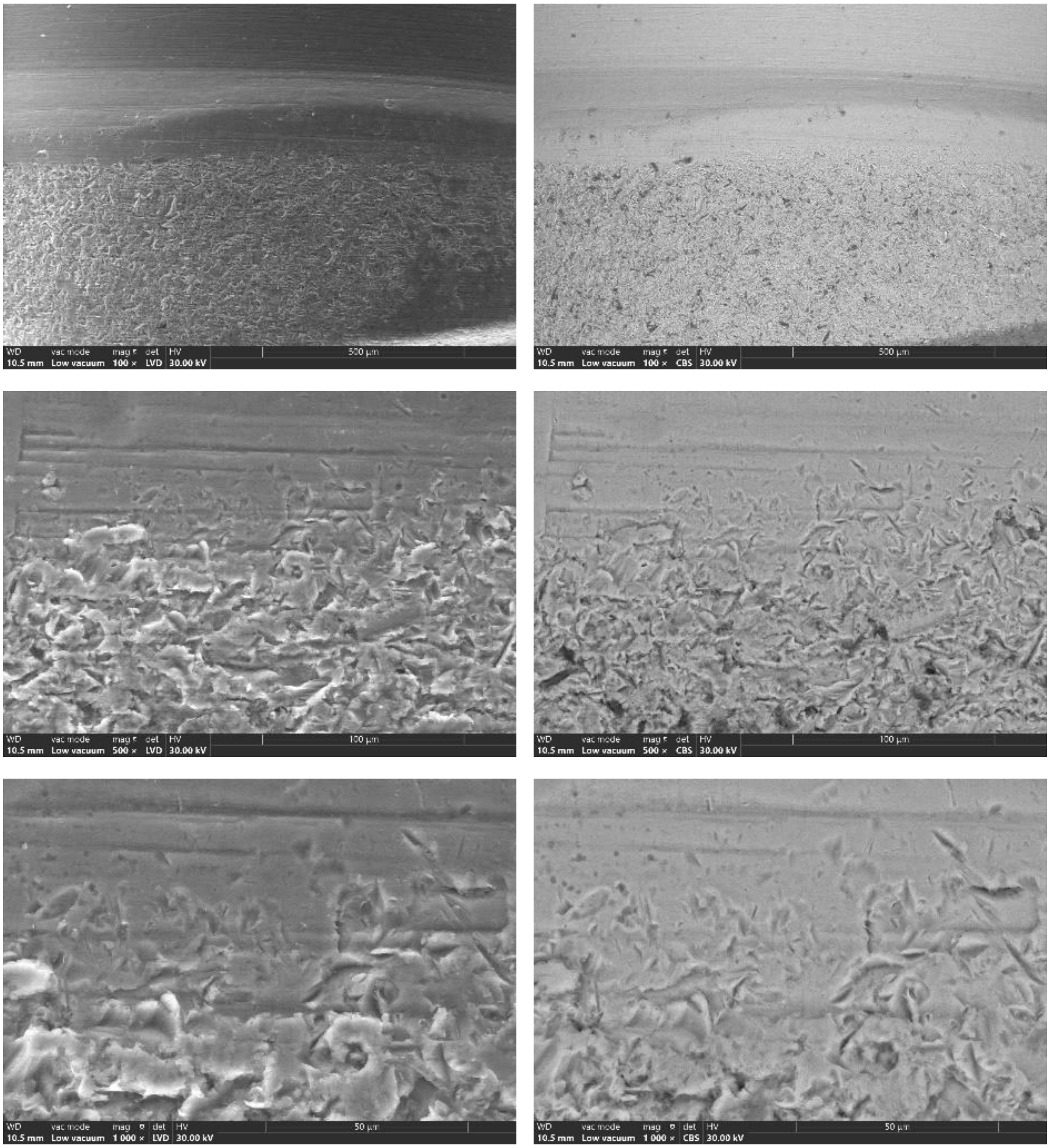


Figure 3.8. Morphology of the reference sample surface (R) in Zone 1.

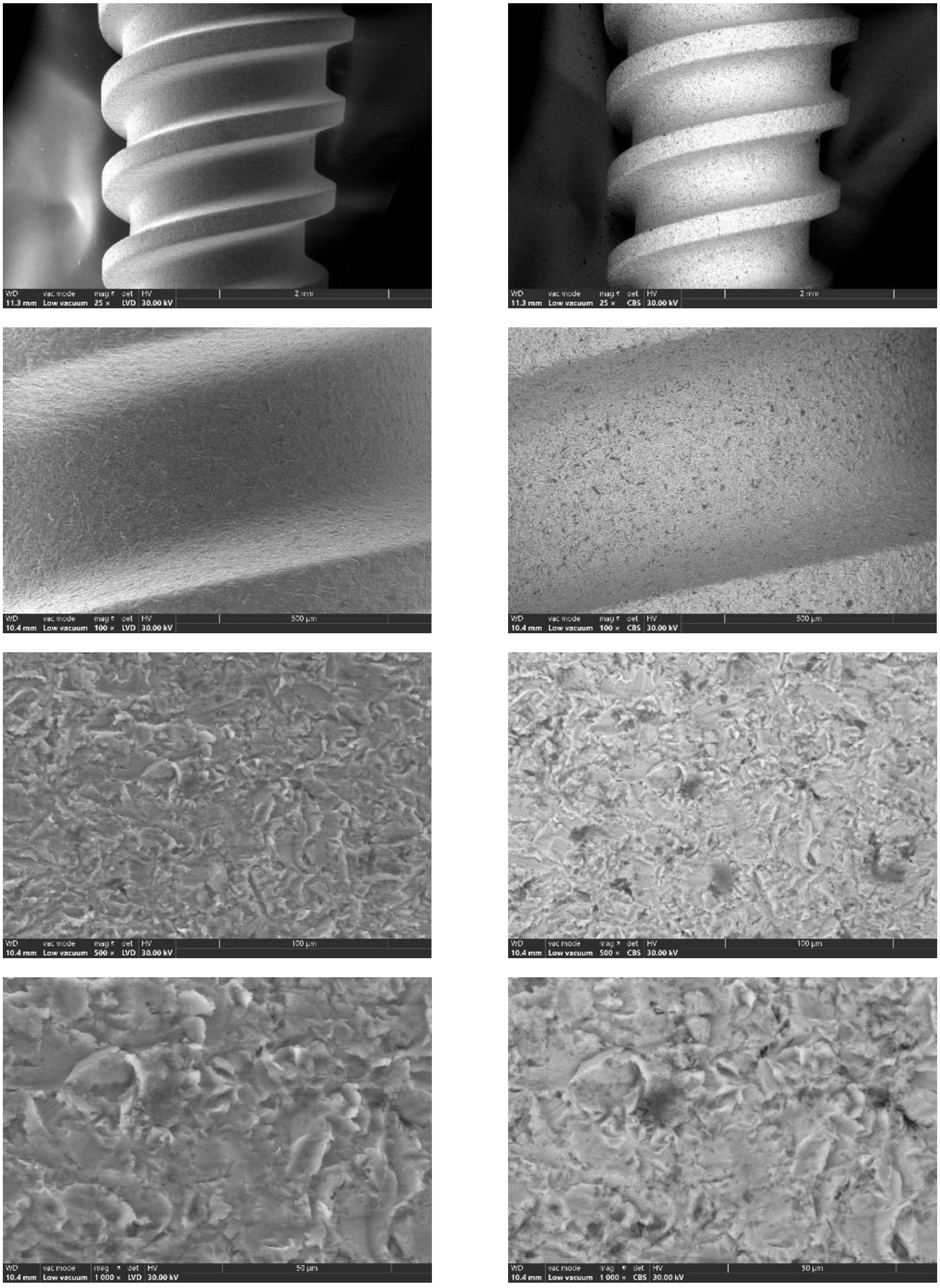


Figure 3.9. SEM images of the reference sample (R), Zone 2 – between the threads of the implant.

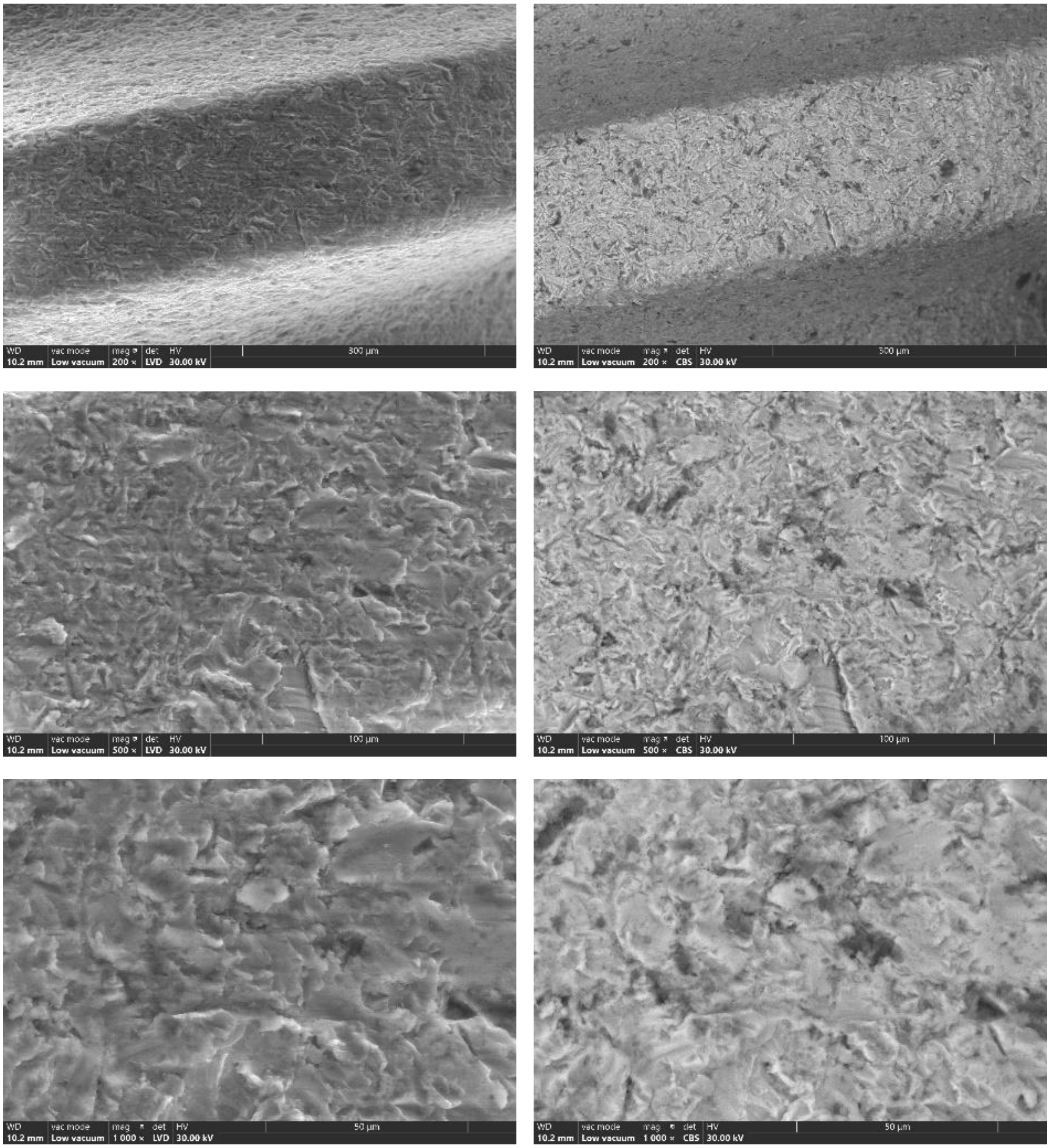


Figure 3.10. Morphology of the reference sample surface (R), on the threads of the implant (Zone 2).

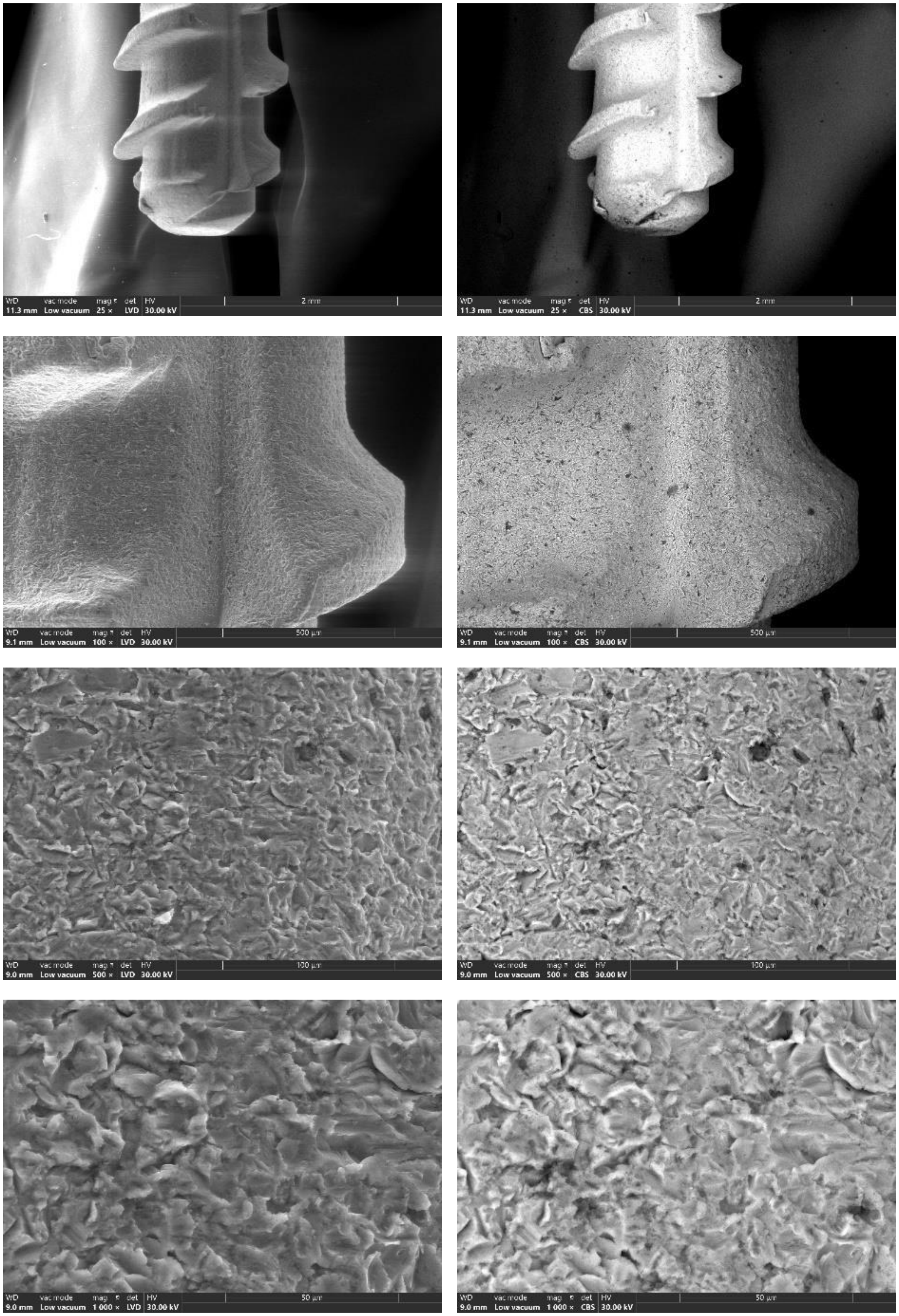


Figure 3.11. Morphology of the reference sample surface (R), in Zone 3.

Rough surfaces contribute to the differentiation of osteoblasts, while smooth surfaces promote the adhesion of fibroblasts. Following the applied treatments, the implant surfaces exhibited a texture characterized by the presence of irregular cavities and alternating peaks, which facilitates the formation of bone tissue.

For compositional characterization, EDS analysis was used to identify the chemical elements present on the implant surface. The results confirmed the presence of titanium (Ti), aluminum (Al), and vanadium (V), which are characteristic of the Ti6Al4V alloy. The analysis performed at three different points highlighted accumulations of oxygen and aluminum in certain areas, indicating that the alumina abrasive particles used in the surface treatments were not completely removed. *(Figure 3.12; Figure 3.13)*

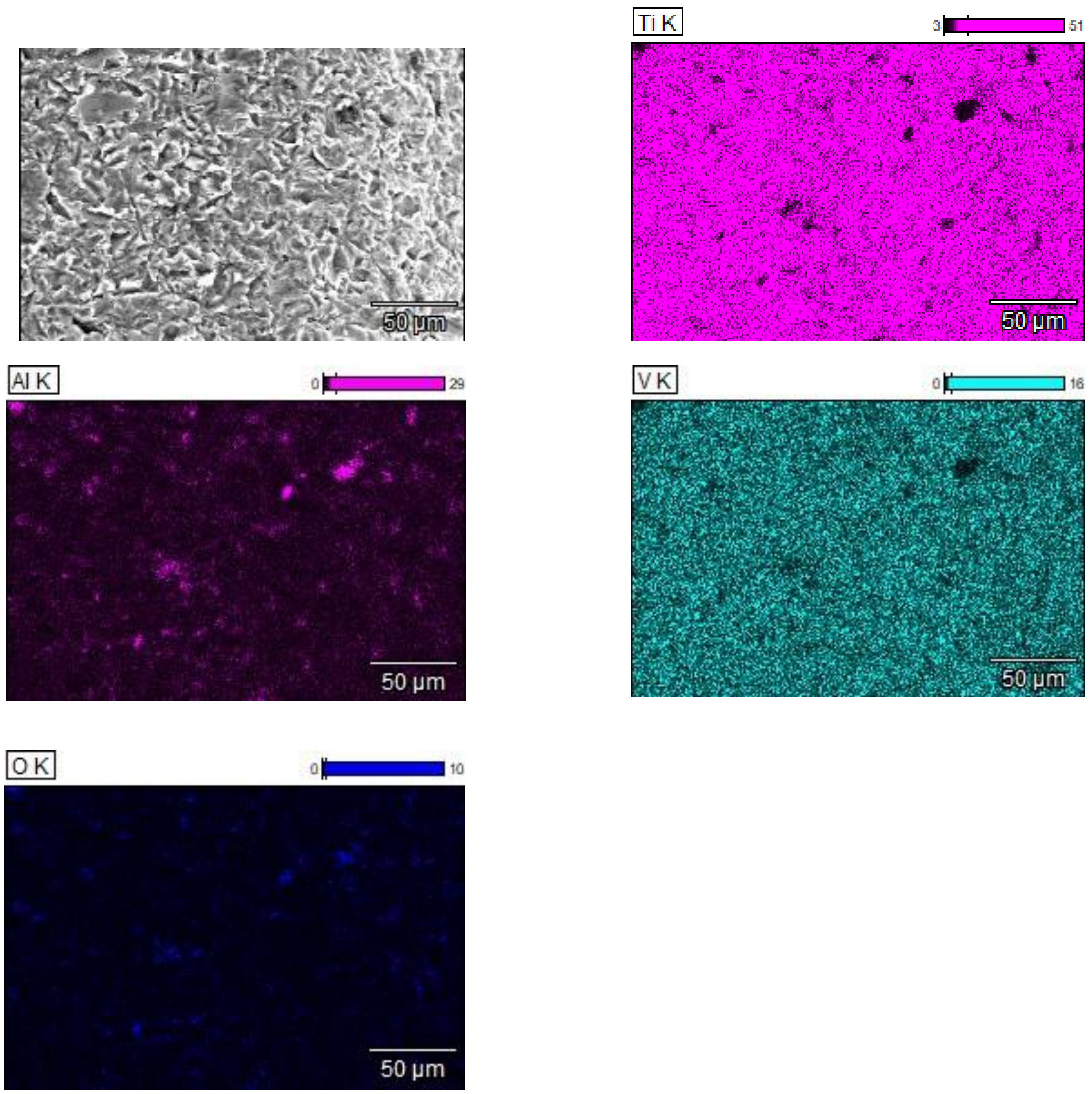
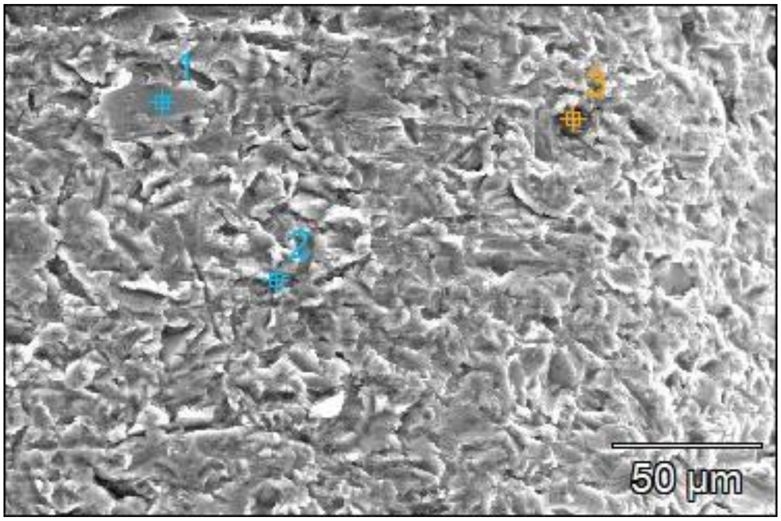


Figure 3.12. Elemental distribution on the surface of the reference sample (R)



	Procente masice (%)			
Puncte	***O***	***Al***	***Ti***	***V***
1	0	7,85±0,08	87,40±0,31	4,75±0,09
2	39,53±0,59	25,75±0,11	32,63±0,14	2,10±0,06
3	54,15±0,32	32,49±0,13	12,78±0,07	0,58±0,04

Figure 3.13. EDS analysis at three points for the reference sample (R).

Evaluation of the surface roughness of the reference sample (R)

The surface roughness of the implant is a determining factor for osseointegration, influencing the interaction between the implant and bone. The roughness parameters analyzed included Ra (average roughness), Rq (root mean square roughness), and Rz (maximum profile height). To obtain a detailed analysis, the reference implant was divided into three distinct zones, and measurements were taken at multiple points extracted between the threads of the implant. *(Figure 3.14; Figure 3.15 – 3.22)*

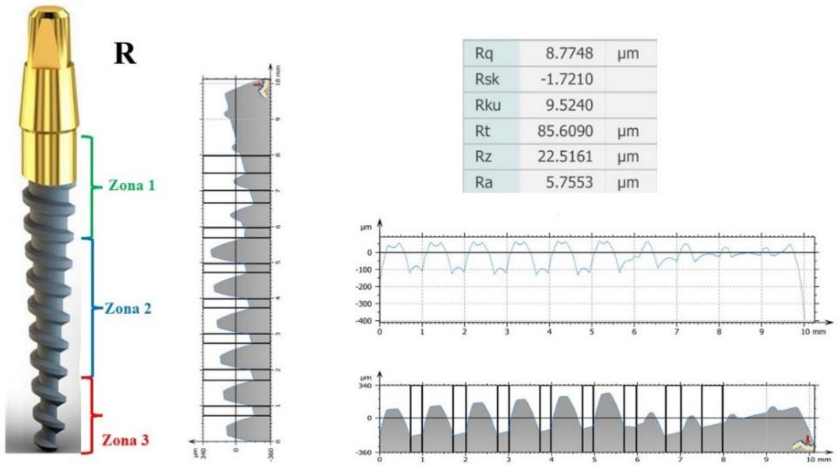


Figure 3.14 presents the determination of the roughness for the reference implant (R) – Side 1. a) Shows the profile of the entire implant R, with the highlighted areas extracted for roughness measurement between the threads. b) Displays the profile of the entire implant R, focusing only on the extracted areas. c) Illustrates the roughness profile of the entire implant R, considering only the extracted zones, along with the resulting parameters.

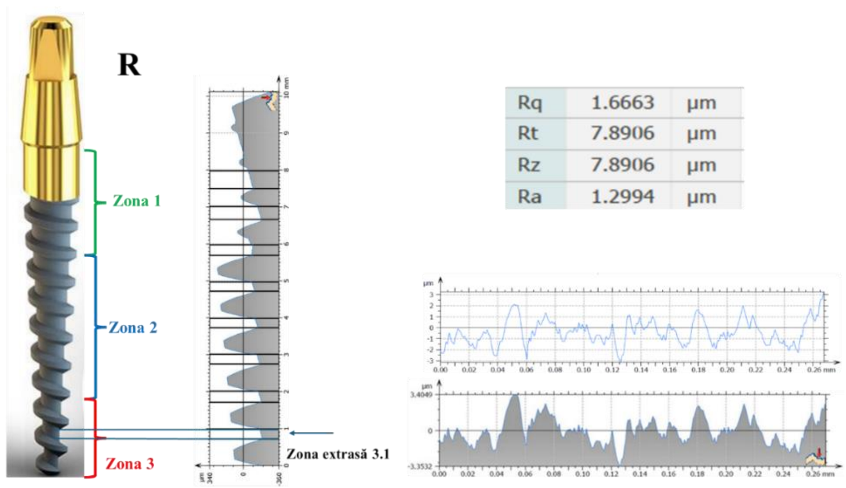


Figure 3.15. Determination of the roughness of the extracted zone 3.1 of implant R, showing the profile of implant R and the resulting parameters

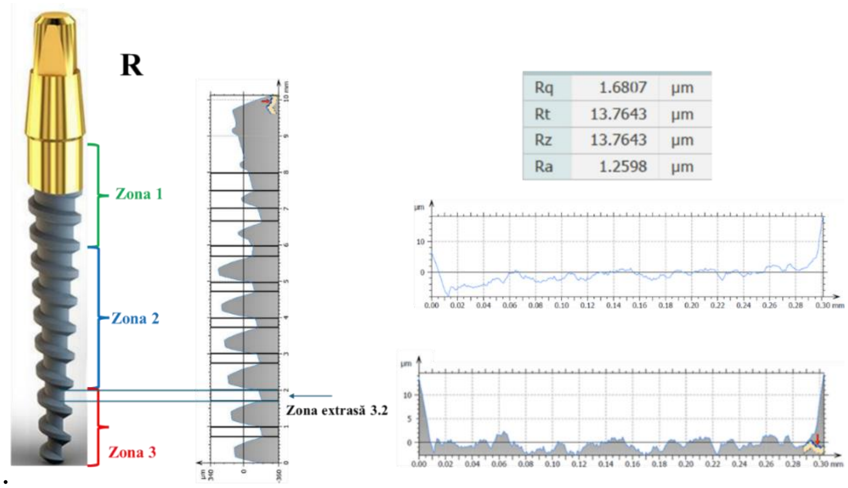


Figure 3.16. Determination of the roughness of the extracted zone 3.2 of implant R, showing the profile of implant R and the resulting parameters.

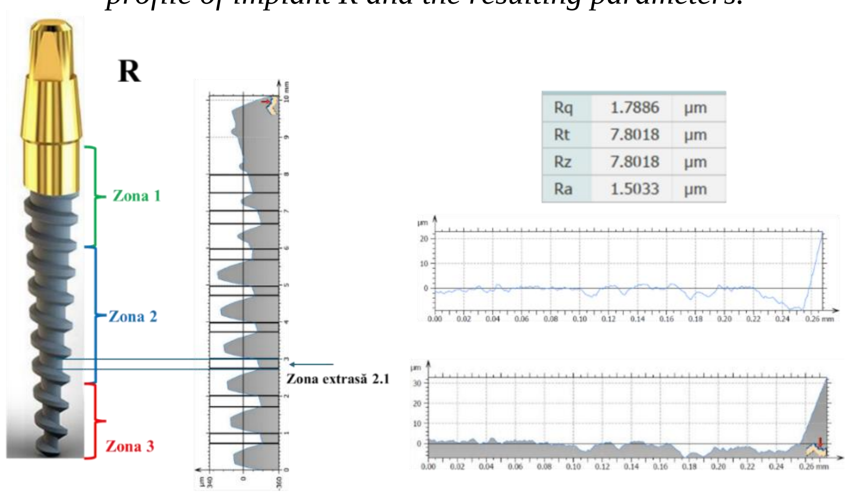


Figure 3.17. Determination of the roughness of the extracted zone 2.1 of implant R, showing the profile of implant R and the resulting parameters.

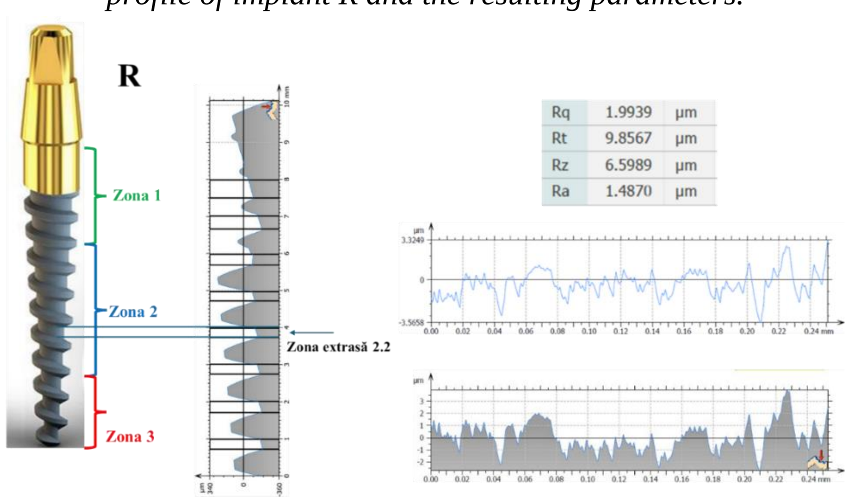
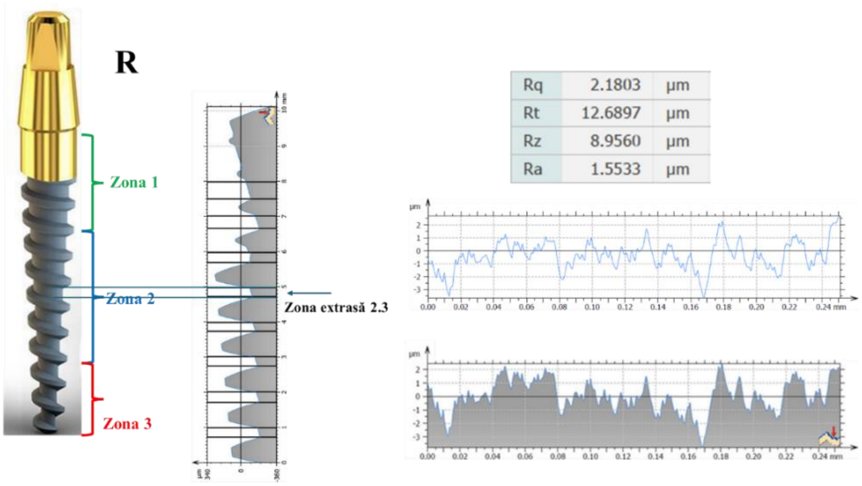


Figure 3.18. Determination of the roughness of the extracted zone 2.2 of implant R, showing the profile of implant R and the resulting parameters



s.

Figure 3.19. Determination of the roughness of the extracted zone 2.3 of implant R, showing the profile of implant R and the resulting parameters.

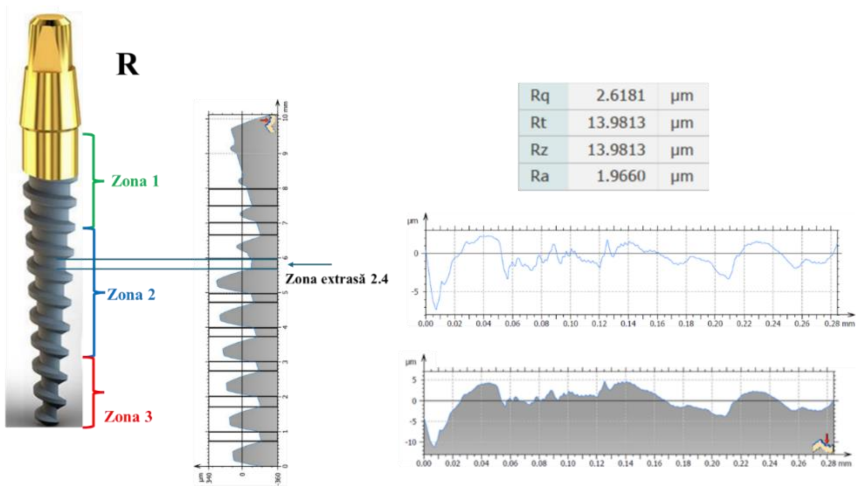


Figure 3.20. Determination of the roughness of the extracted zone 2.4 of implant R, showing the profile of implant R and the resulting parameters.

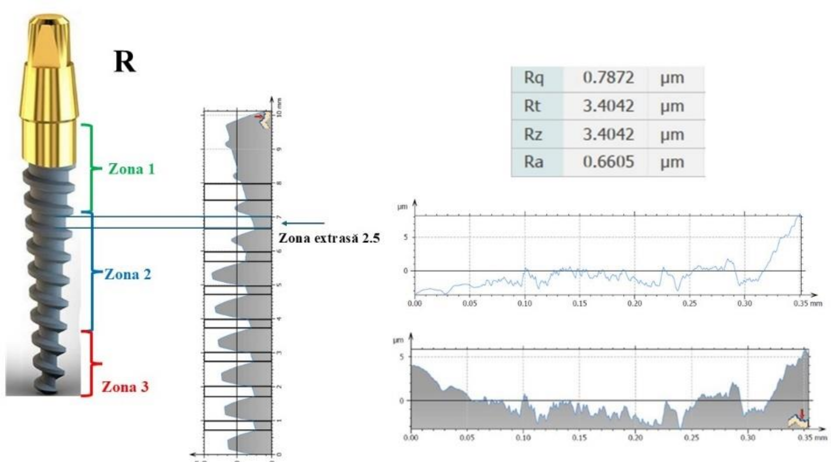


Figure 3.21. Determination of the roughness of the extracted zone 2.5 of implant R, showing the profile of implant R and the resulting parameters

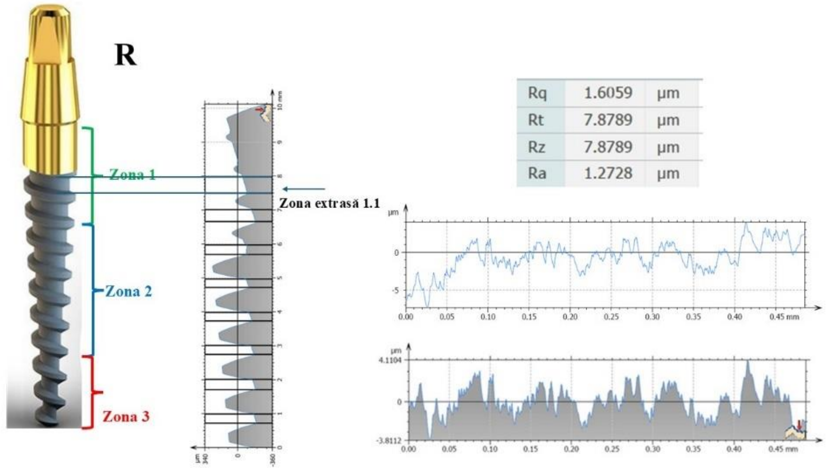


Figure 3.22. Determination of the roughness of the extracted zone 1.1 of implant R, showing the profile of implant R and the resulting parameters.

The values obtained for the roughness parameters were:

Ra – 5,7553 µm

Rq – 8,7748 µm

Rz – 22,5161 µm

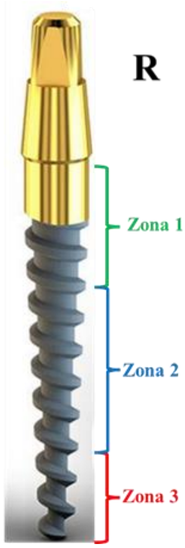
These values indicate a moderately rough surface, optimal for osseointegration, as surfaces with increased roughness provide a larger contact area and enhance cell adhesion.

Evaluation of the wettability of the reference sample surface (R)

Wettability of the dental implant is a crucial parameter, as it influences protein adhesion, cell interaction with the implant, and the formation of bacterial biofilm. The ability of a surface to retain liquid is determined by measuring the contact angle.

- Hydrophilic surfaces (contact angle < 90°) are favorable for osseointegration.
- Hydrophobic surfaces (contact angle > 90°) can slow down the bone integration process.

To determine the contact angle, three testing liquids were used: distilled water, diiodomethane, and ethylene glycol. The measurements were conducted at room temperature and under controlled humidity. The results indicated that the implants analyzed exhibited a hydrophilic behavior, with a contact angle ranging from 56° to 57°, which is favorable for osseointegration. *(Figure 3.27)*



R	Contact Angle (°)			Surface Free Energy (SFE) (mN/m)
	Water	**Diiodomethane**	**Ethylene Glycol**	
Zone 1	56	32	60	45
Zone 2	57	30	62	46
Zone 3	56	32	59	46

Figure 3.27. Values of surface free energy and contact angle for the reference sample (R).

CHAPTER 4. SURFACE MODIFICATION OF DENTAL IMPLANTS THROUGH BIOCERAMIC TREATMENT USING THE ABRASION PROCESS AND CHARACTERIZATION OF THESE IMPLANT SURFACES

This chapter examines the surface modifications of dental implants using bioceramics, specifically hydroxyapatite, through an air abrasion process. The research focuses on the characterization of the modified implant surfaces and their impact on osseointegration.

4.1. Materials and methods

Hydroxyapatite (HA) has the chemical formula $Ca_{10}(PO_4)_6(OH)_2$ and a Ca/P ratio of 1.67, making it an essential compound in the structure of human bone. However, biological apatites significantly differ from stoichiometric hydroxyapatite due to lower crystallinity, smaller crystal sizes, and higher solubility. This variability in mineral composition influences the integration of dental implants into the bone.

To mimic the bone mineral structure, advanced hydroxyapatite coating technologies have been developed for dental implants, aiming to improve osseointegration. Among the most commonly used surface modification methods is air abrasion, which involves the controlled deposition of hydroxyapatite particles on the implant surface. *(Table 4.1)*

Table 4.1. Chemical and structural comparisons between the mineral component of bone, enamel, and dentin, compared to synthetic hydroxyapatite (HA).

Compoziție (% gravimetrice)	HA	Smalț	Dentină	Os
Ca	39,68	36,50	35,10	34,80
P	18,45	17,10	16,90	15,20
Raport molar Ca/P	1,67	1,63	1,61	1,71
Fază anorganică totală	100	97	70	65
Fază organică totală	-	1,50	20	25
Apă	-	1,50	10	10
Caracteristici cristalografice: parametri de rețea (± 0,003 Å)				
a = b (Å)	9,418	9,441	9,421	9,410
c (Å)	6,883	6,880	6,887	6,890
Indice de cristalinitate	100	70 - 75	33 - 37	33 - 37

Research has shown that the use of osteoconductive powders, such as bioglass and hydroxyapatite, contributes to restoring the bioactivity of implant surfaces and enhances the reosseointegration capacity. However, the influence of this treatment on the physicochemical properties of the implant surface and its dissolution behavior has not been sufficiently explored.

This study aims to evaluate air abrasion combined with hydroxyapatite as a method for surface modification of Ti6Al4V alloy dental implants, analyzing how this technique can enhance osseointegration and the implant-bone interaction.

Surface modification of implants through bioceramic treatment using the abrasion process

For the surface treatment of the implants, hydroxyapatite was deposited using abrasion with a specialized device connected to an air compressor. The implants were rinsed with deionized water and dried before the treatment was applied. The process was carried out under two experimental conditions:

⇾ At a pressure of 0.21 MPa and a spraying time of 2 minutes.

⇾ At a pressure of 0.41 MPa and a spraying time of 4 minutes.

Table 4.2 Presents the process parameters for abrasion for each tested sample.

Sample	**Abrasion Process Parameters**	
	Pressure (MPa)	**Time (min.)**
R	**-**	**-**
P1	**0,21**	**2**
P2	**0,41**	**4**

4.2. Modern methods of dental implant surface modification

Various surface treatments are used to enhance osseointegration of dental implants. These modifications can alter the topography, morphology, and chemistry of the implant surfaces. For example, rough surfaces promote osseointegration more effectively than smooth ones. Some of the methods used include:

- Sandblasting with titanium dioxide (TiO_2) particles is used to modify the surface roughness of dental implants.

- Acid etching is used to improve the adhesion of bone cells to the implant surface..

- Anodic oxidation increases the thickness of the titanium oxide layer on the implant surface and improves its interaction with bone tissue.

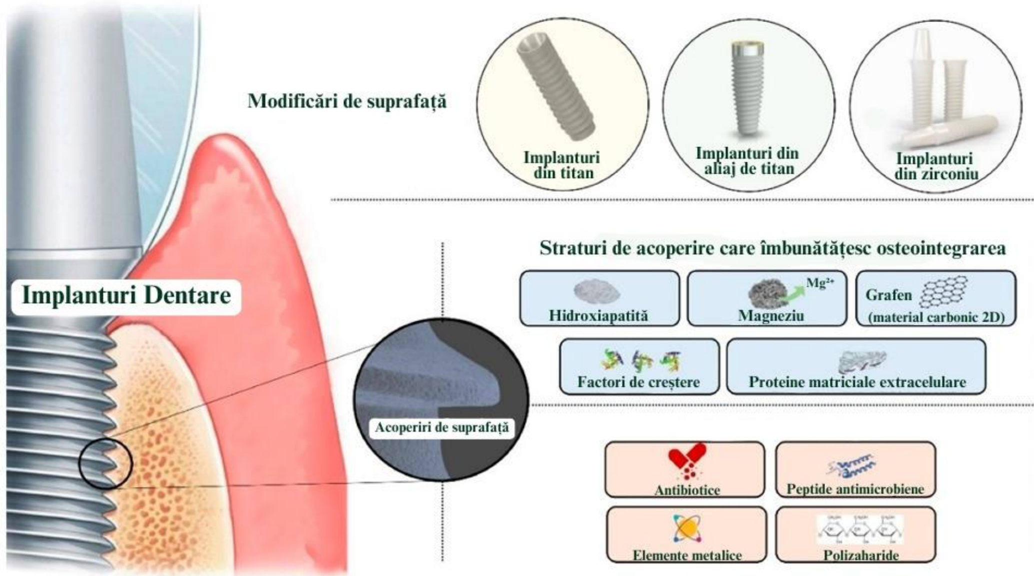


Figure 4.1. Schematic representation of the modified surfaces and functional coatings of dental implants. [3]

Surface chemistry modification of implants

In addition to mechanical treatments, the surface chemistry of implants can be modified through specific chemical processes, such as acid treatments, alkaline treatments, and the use of hydrogen peroxide. These modifications influence the biocompatibility and osseointegration of the implants. *(Figure 4.2)*

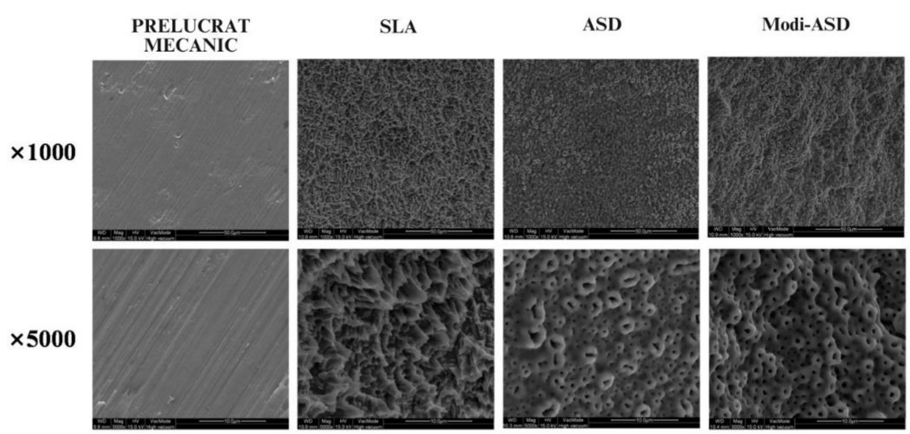


Figure 4.2. SEM images of titanium substrates with modified surfaces.. SLA: Sandblasted and acid-etched Ti, ASD: Anodized Ti, Modi-ASD: Sandblasted/acid-etched and anodized Ti [4]

4.3. Experimental results and discussions

This subsection analyzes the results obtained from the surface treatment of titanium dental implants with hydroxyapatite, aimed at improving osseointegration. To characterize the treated surfaces in detail, various investigation methods were used, including Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), chemical composition analysis by EDS, and the determination of surface roughness and wettability. These analyses allowed the evaluation of the morphology, crystalline structure, and chemical properties of the implant surfaces, comparing the results obtained for the samples treated under different experimental conditions.

To determine the structure and particle size of the hydroxyapatite used in the surface modification process of the implants, investigations were conducted using Scanning Electron Microscopy (SEM). The analyses revealed that the hydroxyapatite powder consists of aggregates of fine crystals with dimensions ranging from 25-29 nm, which are impossible to observe individually in the SEM images. Additionally, the compositional analysis through Energy Dispersive X-ray Spectroscopy (EDS) confirmed the presence of calcium (Ca) and phosphorus (P), essential elements for osseointegration of the implant. *(Figure 4.22)*

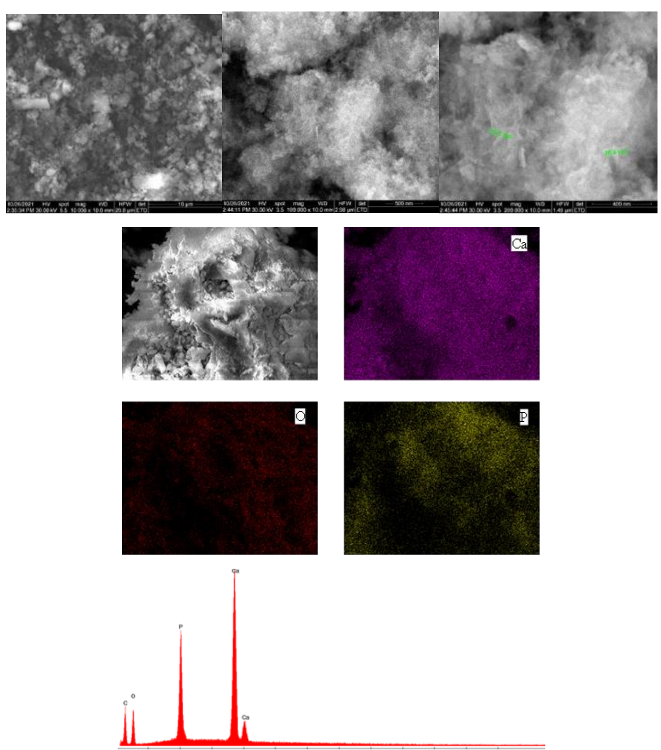


Figure 4.22. SEM images and EDS mapping for hydroxyapatite powder.

Crystallographic structure of hydroxyapatite

To determine the crystalline phases present in hydroxyapatite, X-ray Diffraction (XRD) was used. The results indicated a polycrystalline structure characteristic of $Ca_{10}(PO_4)_6(OH)_2$ hydroxyapatite, with distinct peaks at 2ϴ = 25.7°, 31.85°, and 32.98°. In addition to hydroxyapatite, the analysis also revealed a minor phase of monocalcium phosphate ($CaHPO_4$), suggesting the presence of additional compounds that may influence the implant's behavior in the biological environment. *(Figure 4.23)*

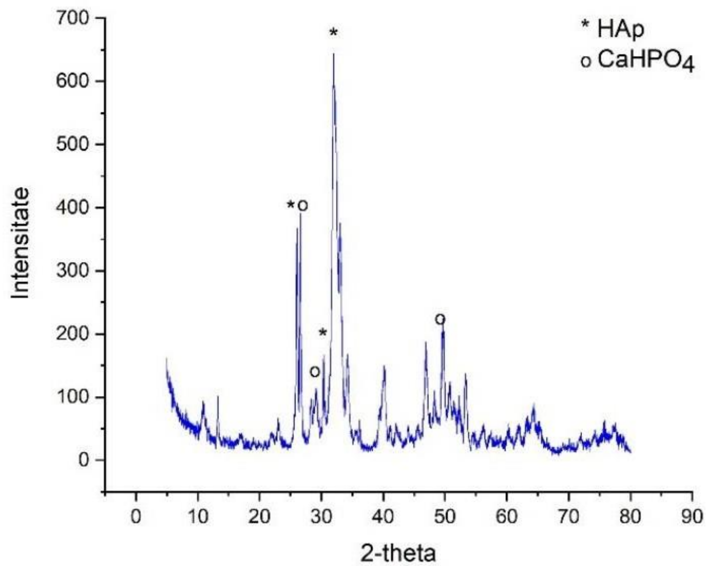


Figure 4.23. X-ray diffraction pattern for HA (hydroxyapatite).

Analysis of functional groups through FT-IR spectroscopy

To confirm the chemical structure of hydroxyapatite, Fourier Transform Infrared Spectroscopy (FT-IR) was used. The obtained spectrum highlighted the presence of specific phosphate (P-O) group vibrations at ~560 cm^{-1}, ~604 cm^{-1}, and ~1015 cm^{-1}, as well as bands corresponding to hydroxyl (OH) groups at ~630 cm^{-1}, thereby confirming the mineral nature of the material deposited on the implant surface. *(Figure 4.24)*

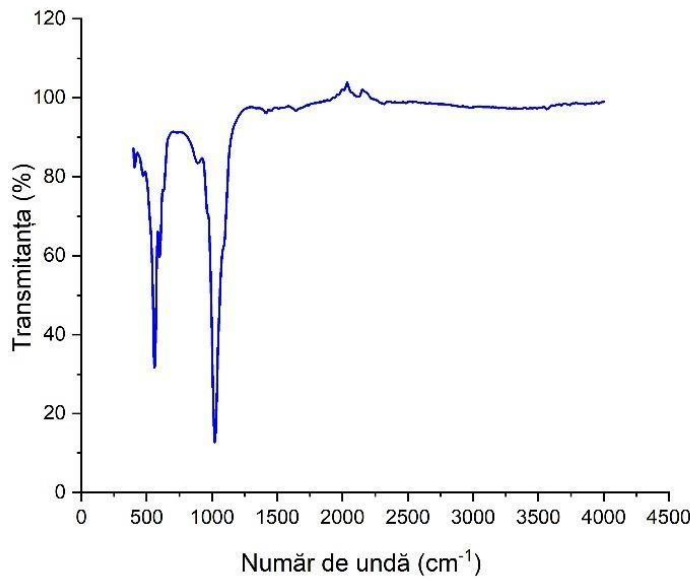


Figure 4.24. FT-IR spectrum for HA (hydroxyapatite)

Evaluation of the morphology of treated implant surfaces

Scanning Electron Microscopy (SEM) was used to investigate the changes occurring on the implant surfaces after the hydroxyapatite treatment. The analyses were conducted for two experimental samples, P1 and P2, treated under different pressure conditions and exposure times during the abrasion with hydroxyapatite.

The results for sample P1, treated at lower pressure and shorter exposure time, indicated an uneven distribution of hydroxyapatite, with areas where the deposited material layer was incomplete. *(Figures 4.25 – 4.28)*

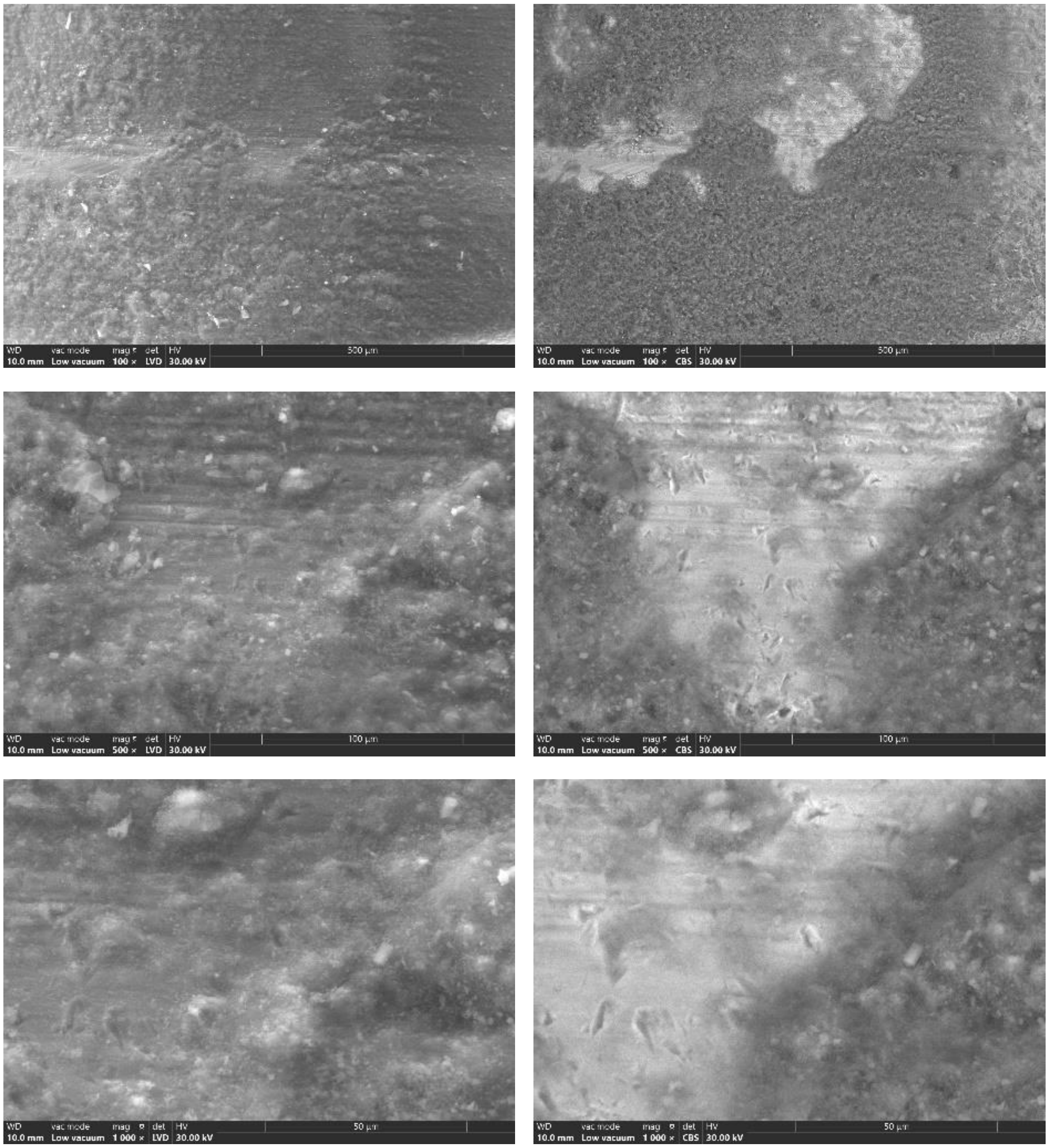


Figure 4.25. Morphology of the surface of sample P1, in Zone 1.

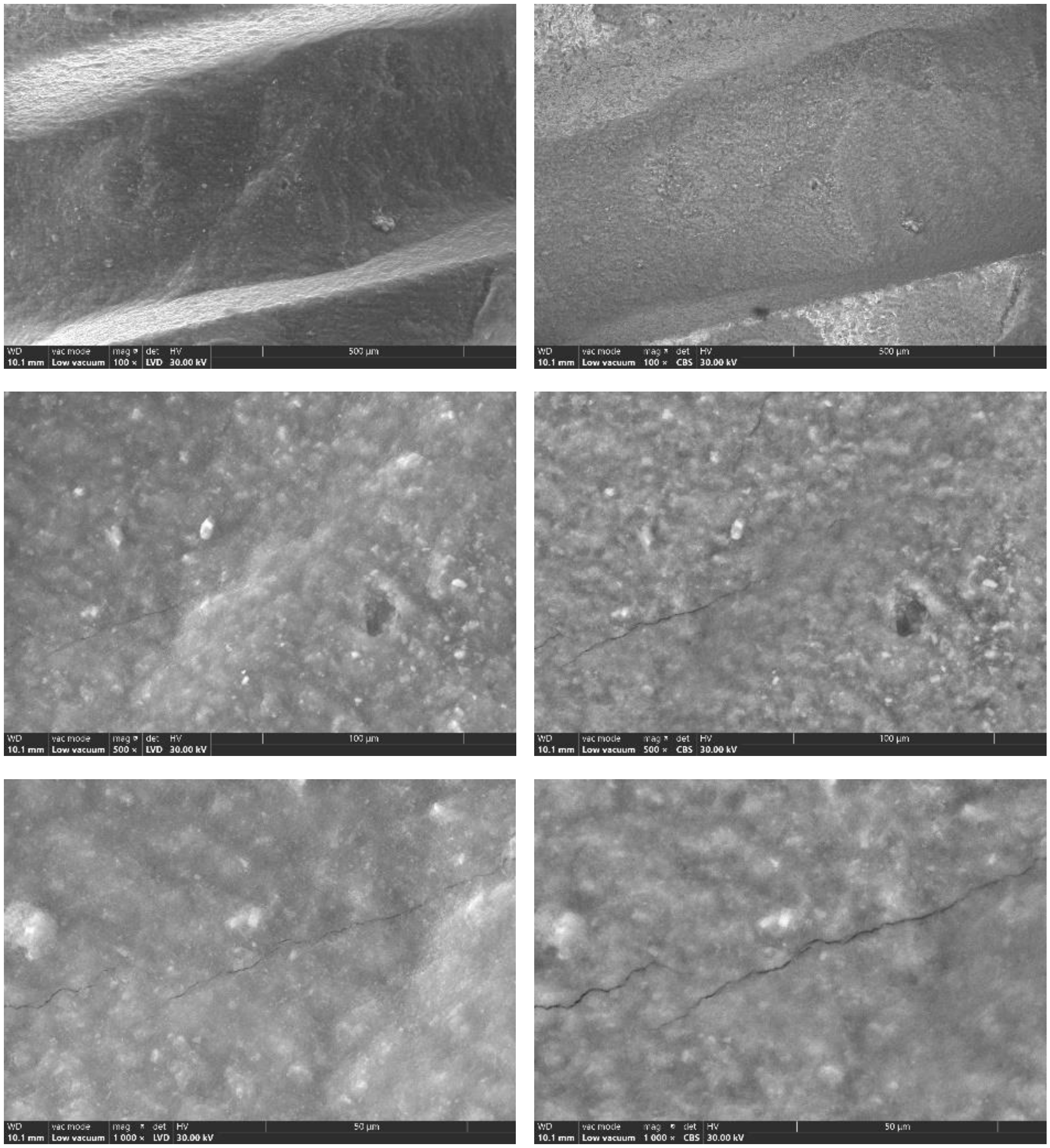


Figure 4.26. Morphology of the surface of sample P1, in Zone 2 – between the threads of the implant.

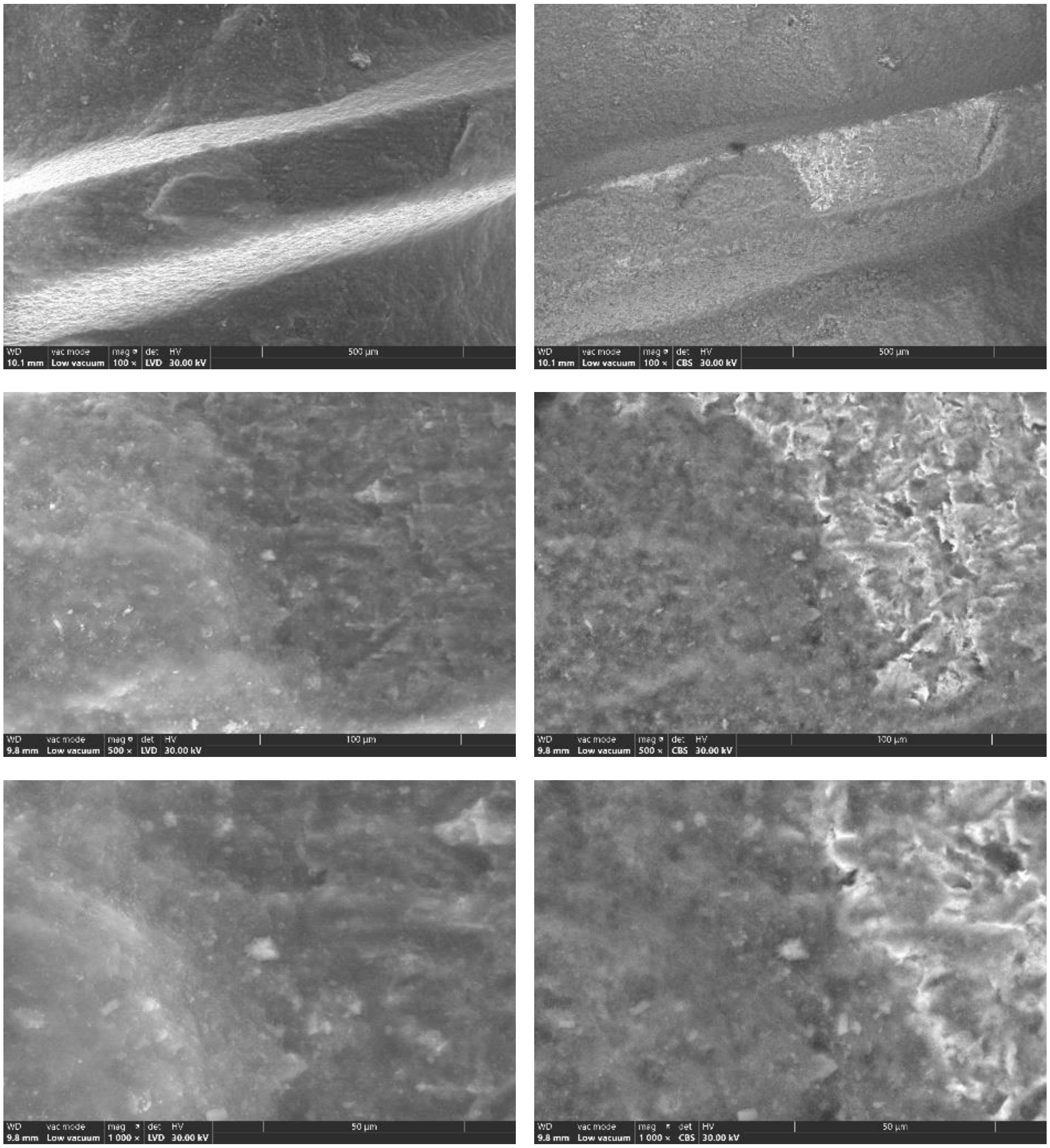


Figure 4.27. Morphology of the surface of sample P1, in Zone 2 – on the threads of the implant.

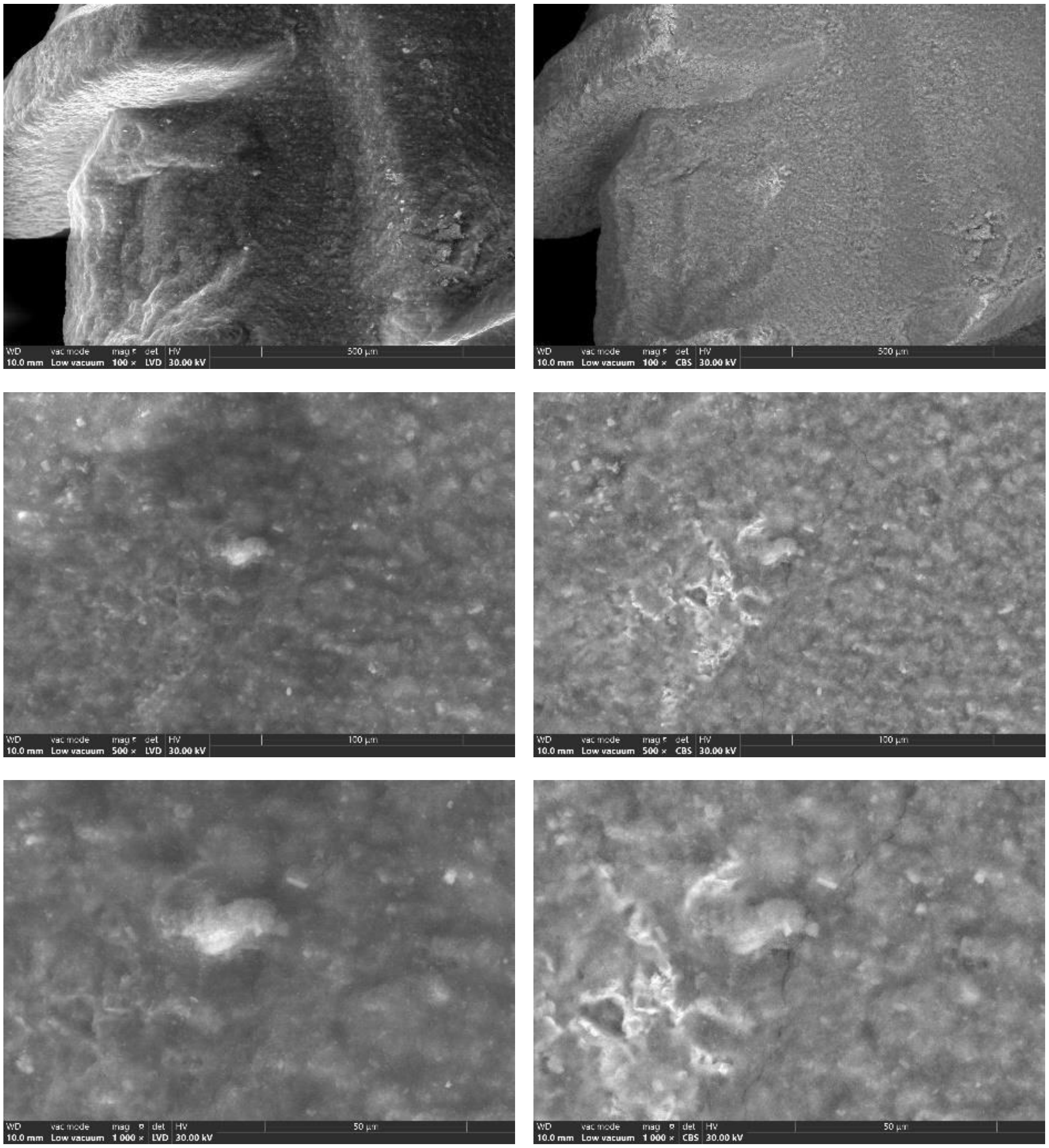


Figure 4.28. Morphology of the surface of sample P1, in Zone 3.

From a different standpoint, sample P2, treated at higher pressure and with a longer exposure time, exhibited a more homogeneous layer, suggesting that the treatment parameters significantly influence the distribution of hydroxyapatite on the implant surface. *(Figures 4.31 – 4.34)*

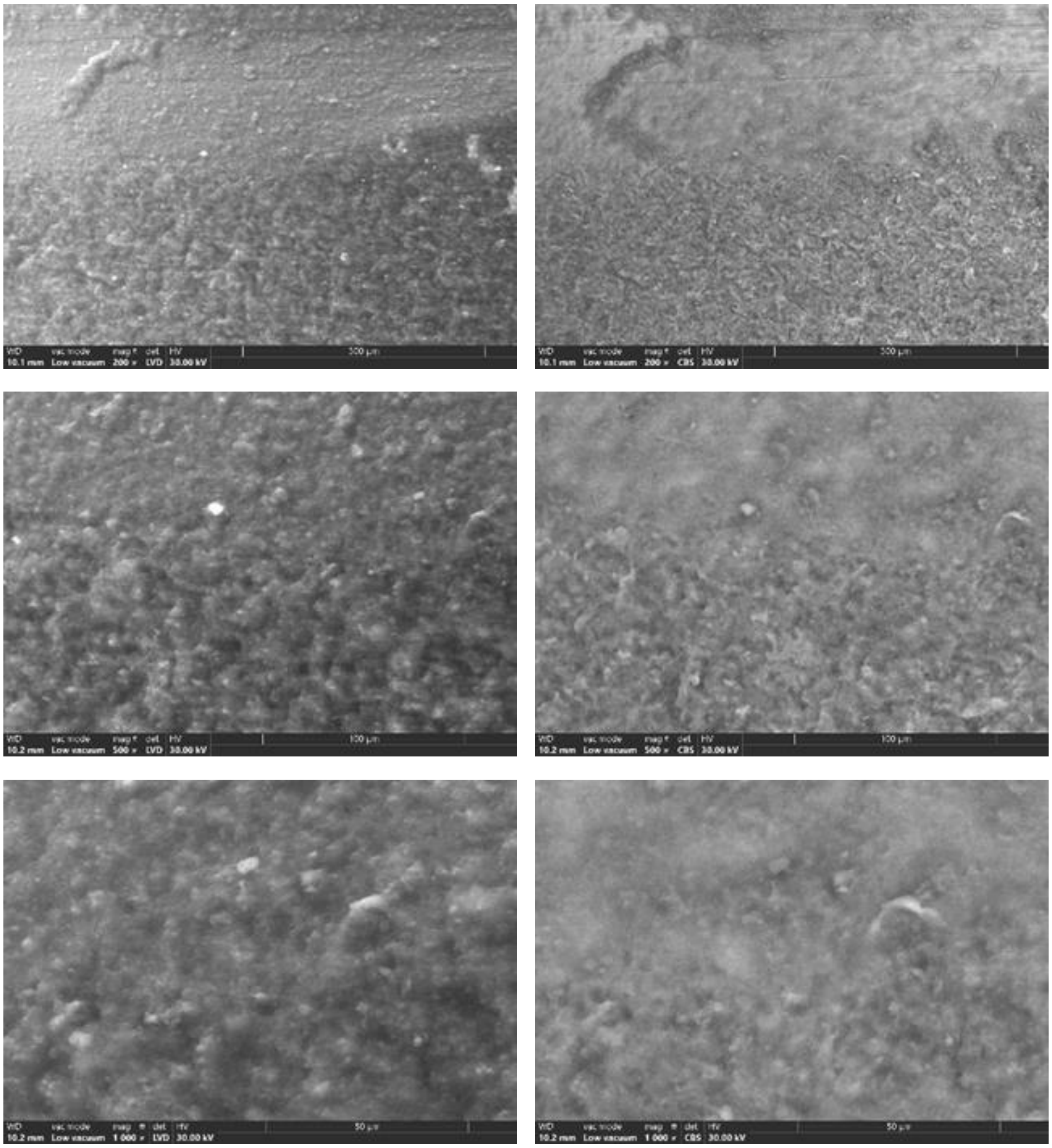


Figure 4.31. Morphology of the surface of sample P2, in Zone 1.

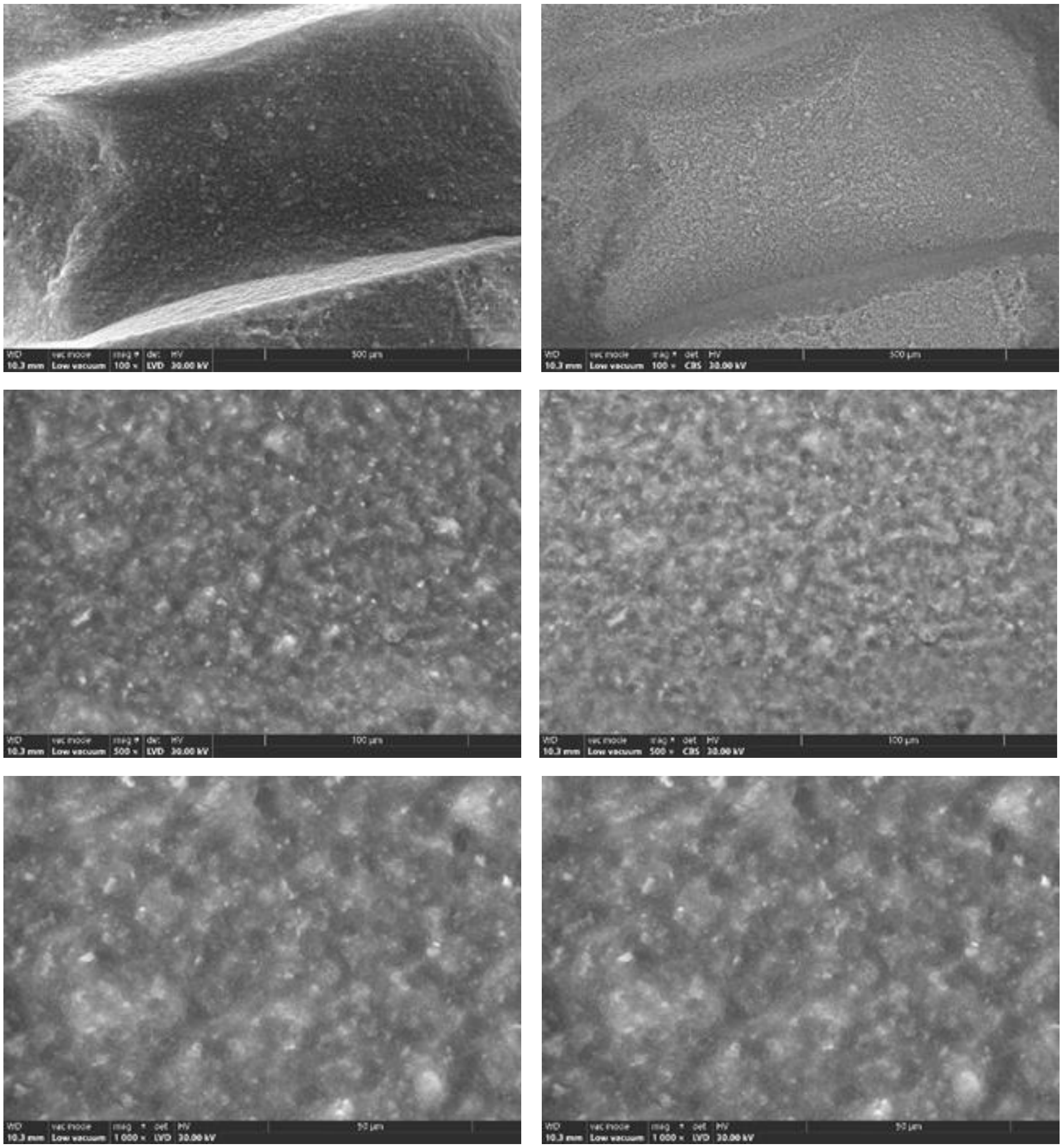


Figure 4.32. Morphology of the surface of sample P2, in Zone 2 – between the threads of the implant.

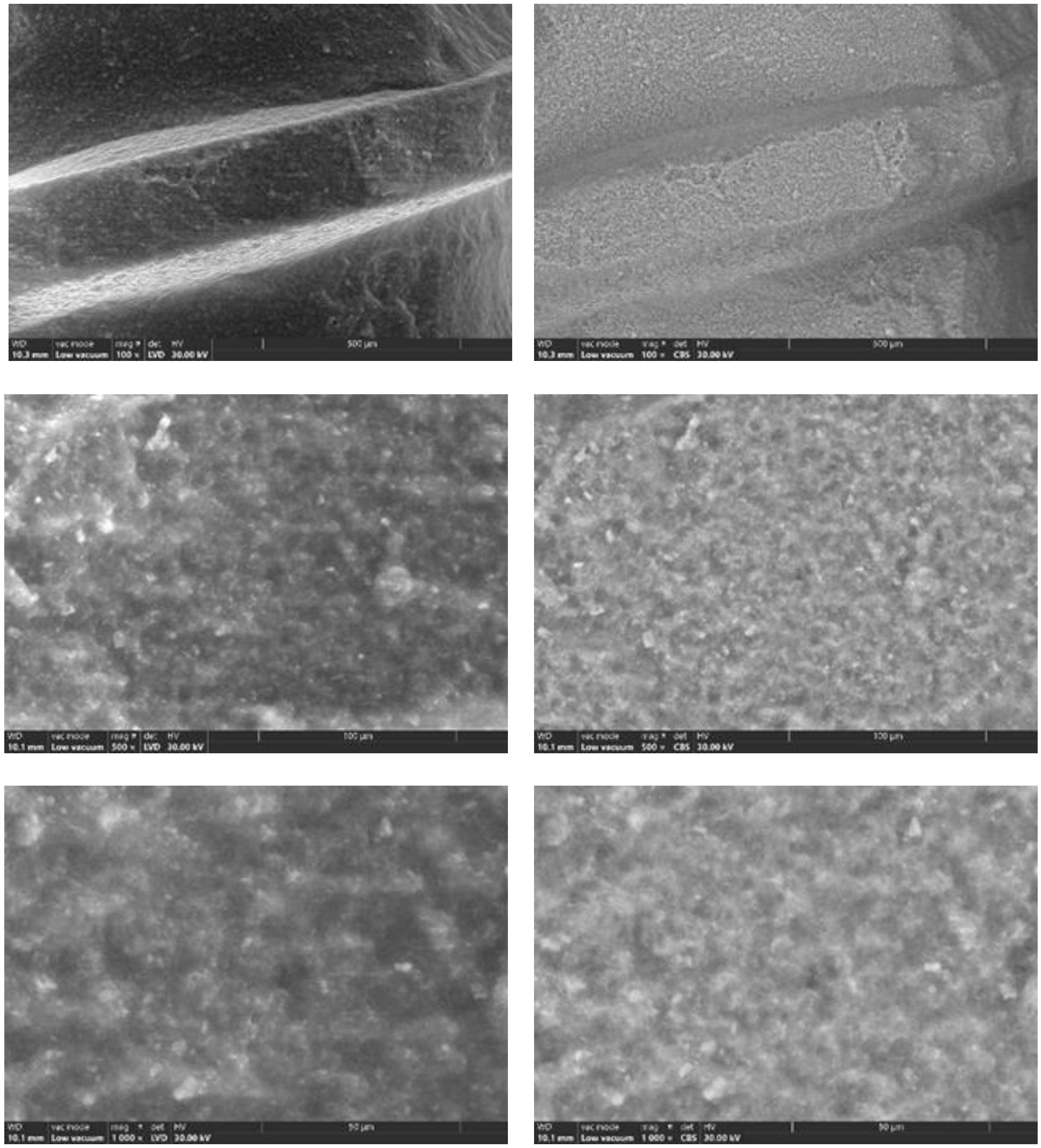


Figure 4.33. Morphology of the surface of sample P2, in Zone 2 – on the threads of the implant

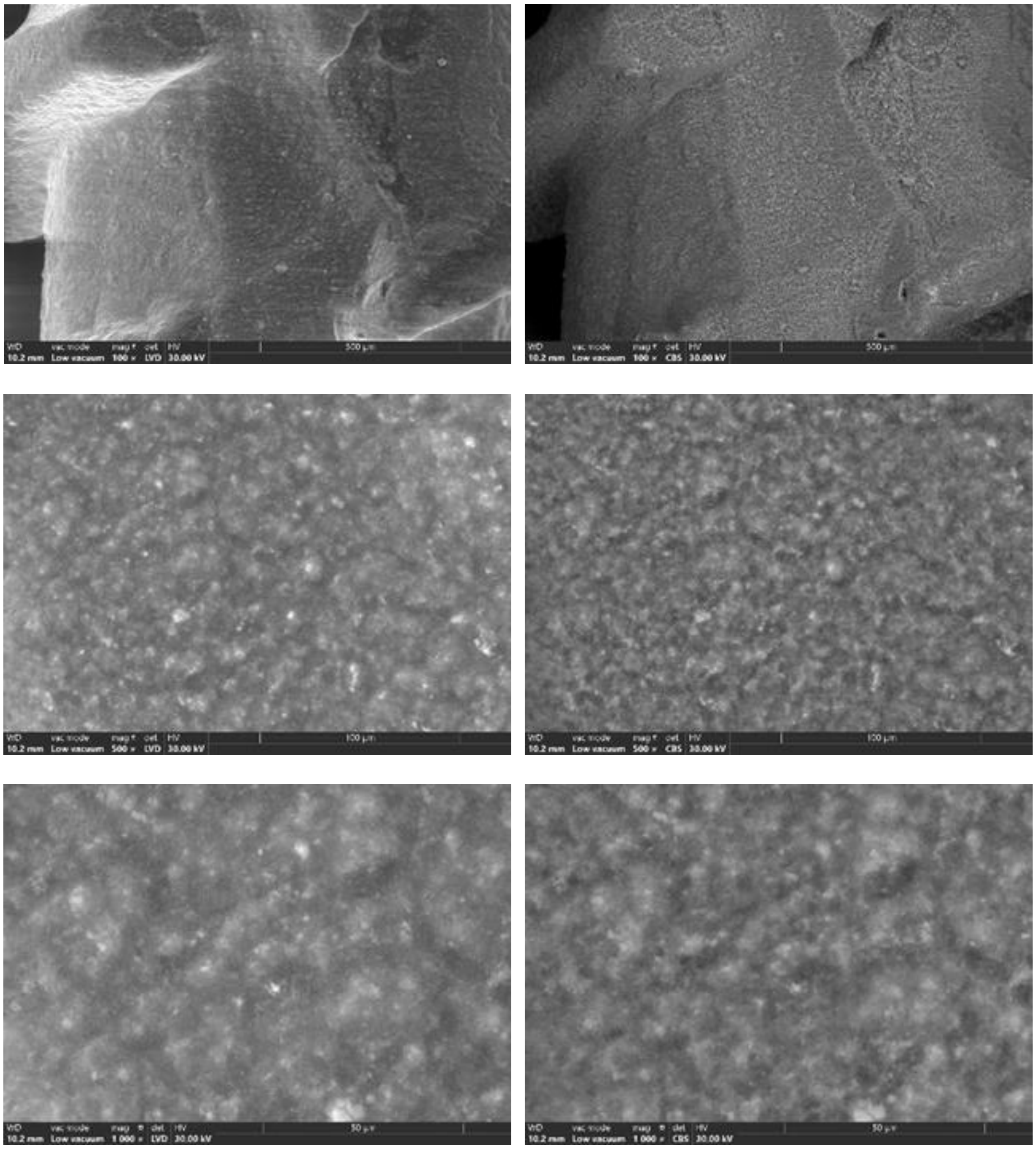


Figure 4.34. Morphology of the surface of sample P2, in Zone 3

Evaluation of the surface roughness of experimental samples P1 and P2

To determine the impact of the treatment on the surface roughness of the implants, measurements were taken for the parameters Ra (average roughness), Rq (root mean square roughness), and Rz (maximum profile height).

Sample P1 exhibited a high roughness (Ra = 12.2322 µm), indicating a more textured surface. Sample P2 had a lower roughness (Ra = 6.185 µm), suggesting a more uniform layer and better adhesion of hydroxyapatite.

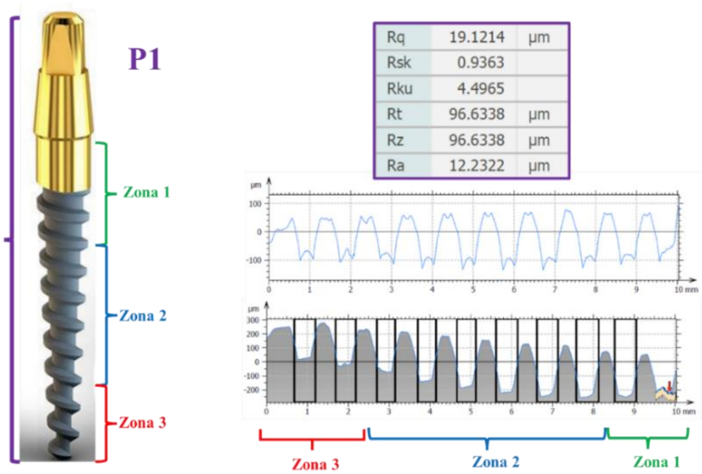


Figure 4.37. Roughness profile of experimental sample P1, highlighting the extracted areas for roughness measurement between its threads and the resulting parameters.

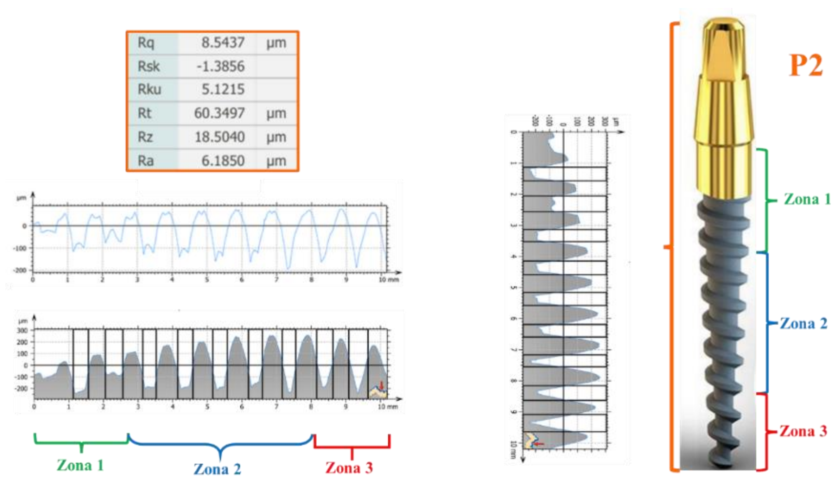


Figure 4.46. Roughness profile of experimental sample P2, highlighting the extracted areas for roughness measurement between its threads and the resulting parameters.

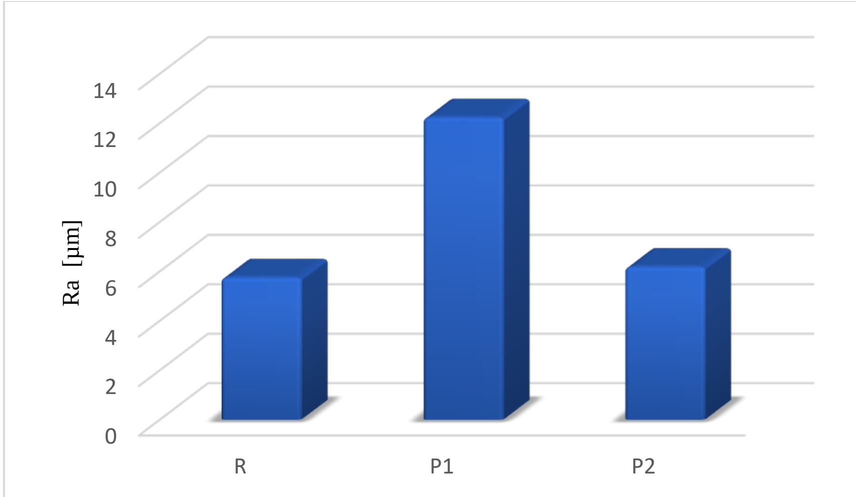


Figure 4.55. Evaluation of the Ra parameter determined on the surface of the implants (R, P1 and P2).

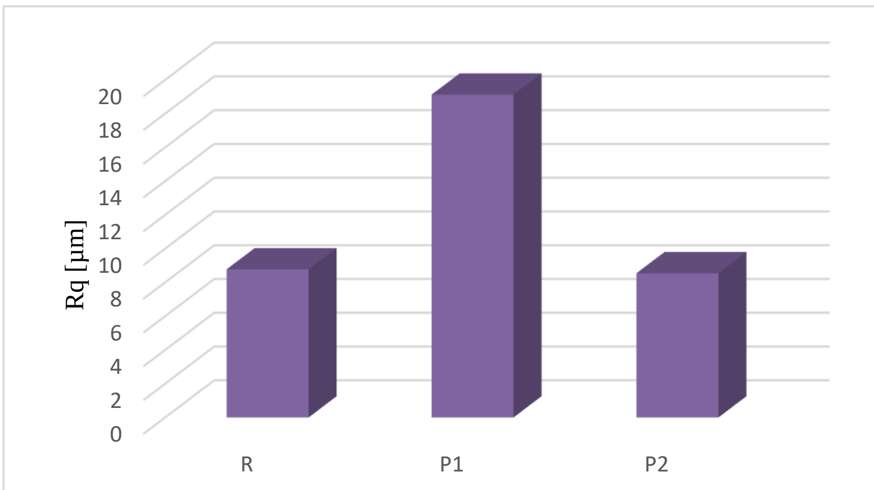


Figure 4.56. Evaluation of the Rq parameter determined on the surface of the implants (R, P1 and P2).

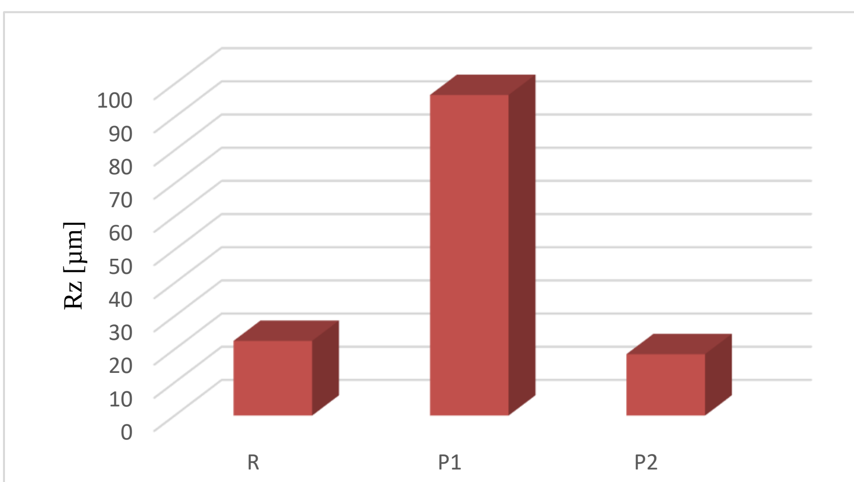
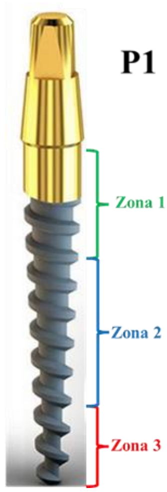


Figure 4.57. Evaluation of the Rz parameter determined on the surface of the implants (R, P1 and P2).

Determination of contact angle and wettability of the surface

Another essential parameter analyzed was the contact angle, which provides information about the wettability of the implant surfaces.

- Sample P1 exhibited a contact angle of 43-44°, indicating moderate wettability. *(Figure 4.62)*

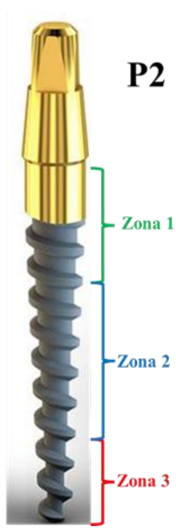


P1

	Contact Angle (°)			Surface Free Energy (SFE) (mN/m)
	Water	Diiodomethane	Ethylene Glycol	
Zone 1	44	39	58	48
Zone 2	43	37	57	49
Zone 3	43	37	57	49

Figure 4.62. Values of surface free energy and contact angle for sample P1.

- Sample P2, with a more uniform hydroxyapatite layer, exhibited a contact angle of 26-28°, indicating a more hydrophilic surface, favorable for rapid osseointegration. *(Figure 4.63)*



P2

	Contact Angle (°)			Surface Free Energy (SFE) (mN/m)
	Water	Diiodomethane	Ethylene Glycol	
Zone 1	28	38	31	51
Zone 2	26	36	29	53
Zone 3	26	35	29	53

Figure 4.63. Values of surface free energy and contact angle for sample P2.

CHAPTER 5. CONCLUSIONS, ORIGINAL CONTRIBUTIONS, AND FUTURE DIRECTIONS

5.1 Conclusions

In relation to current concerns in scientific research within the field of materials science and engineering, aiming to provide new solutions for medicine—both in terms of new materials and processing techniques, including surface modifications—this doctoral thesis makes a significant contribution to the direction of surface modifications applied to dental implants made from Ti6Al4V titanium alloy.

There is a major national and international concern regarding the development of dental implants with modified surfaces that enable better osseointegration and clinical biofunctionality, as highlighted by numerous studies, research publications, and scientific research contracts.

In this context, this doctoral thesis successfully contributes to the analysis of current studies and ongoing scientific research concerning the development of dental implants with modified surfaces.

The conclusions drawn from the experimental studies and research conducted are summarized as follows:

- A comprehensive synthesis of the scientific documentation was carried out, leading to the acquisition of valuable information for establishing an innovative and original experimental program.
- The beneficial role of surface modification of dental implants made from titanium and Ti6Al4V using hydroxyapatite was identified.
- The morphological and structural characterization of the hydroxyapatite particles used to coat the surface of dental implants was performed through scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (EDS), Fourier-transform infrared spectroscopy (FT-IR), and X-ray diffraction (XRD). The bioceramic particles, in the form of agglomerates composed of fine crystallites that cannot be individually observed in micrographs due to their small sizes ranging from 25-29 nm, consist of a major HA phase and a minor $CaHPO_4$, monocalcium phosphate phase.
- It was identified that air abrasion systems are present in most dental offices and are frequently used in clinical practice.

- Based on these theoretical studies, an innovative method for modifying the surface of dental implants made of titanium and Ti6Al4V with the help of hydroxyapatite was proposed, using an air abrasion system for hydroxyapatite coatings. **Practically, hydroxyapatite particles are sprayed onto the surface of the Ti6Al4V dental implant using an air abrasion system with hydroxyapatite particles before the implantation surgery.**
- Since process parameters are particularly important and can affect the final experimental results, two surface modification treatment variants were proposed, resulting in two different types of experimental samples (P1, P2).

Based on the information derived from the specialized literature, the surface properties of dental implants are crucial for their proper osseointegration and biofunctionality. Therefore, the experimental research presented in this doctoral thesis focused on the use of modern methods to determine surface properties, evaluating morphology, elemental composition, roughness, and wettability of the surfaces. Both the reference sample (R), i.e., the commercial Ti6Al4V dental implant, and the samples obtained as a result of the experimental surface modification treatments (P1, P2) were analyzed using SEM-EDS for morphological characterization and elemental composition determination at their surface. Wettability was assessed by determining the contact angle and surface free energy; surface roughness was evaluated by determining the Ra, Rq, and Rz parameters.

The conclusions of the experimental research conducted are presented as follows:

- The effects of surface treatments applied to the commercial dental implants used as a reference (Al2O3 blasting and acid etching) are visible and result in surfaces with similar roughness in all three studied areas of interest, characterized by the presence of irregular cavities alternated uniformly with peaks and valleys.
- Scanning electron microscope images revealed a more uniform distribution of hydroxyapatite on the surface of the dental implant in the case of P2 samples compared to P1 samples. Due to different operating parameters, the air abrasion process with hydroxyapatite particles induces different morphologies on the surface of the Ti6Al4V alloy implant. The presence of a more uniformly distributed hydroxyapatite layer on the surface of the implant will generate a roughness, hydrophilicity, and surface free energy more favorable to the osseointegration process.

- Wettability evaluation revealed an increase in hydrophilicity and surface free energy for the experimental samples with an increase in the deposition time and pressure of hydroxyapatite, indicating better osseointegration of the implant. From this perspective, Surface Treatment 2 generated better results.
- The roughness of a dental implant can be explained by surface texture parameters such as Ra (average roughness) and Rq (root mean square roughness). The Ra value is the arithmetic average of the absolute deviations of the surface profile from the mean line, providing a measure of the overall surface roughness. In the case of the R sample, the Ra value determined across the entire dental implant is 5.75 μm, suggesting that the surface is relatively rough. This roughness was achieved through surface treatments (alumina blasting and acid etching) to improve the implant's osseointegration (the implant's ability to bond with bone tissue).
- The Rq value represents the square root of the average squared deviations of the surface profile and offers a more sensitive indication of surface variations, as it squares the deviations before averaging them.
- The roughness of dental implant surfaces is critical because the higher the roughness, the better the osseointegration. Rough surfaces provide a larger surface area and better mechanical interconnection between the implant and bone, promoting faster and more efficient bone growth. Treatments such as blasting and acid etching are commonly used to modify the implant surface and achieve these roughness values. These methods can result in ideal micrometric-scale roughness for bone-implant interaction.
- Based on the roughness results (Ra and Rq) for the three experimental samples, several conclusions can be drawn regarding the effect of the surface modification conducted in this doctoral thesis on the surface characteristics.
- The P1 samples, whose surface was modified at low pressure for 2 minutes, have a higher average roughness value than the reference sample. This indicates that applying hydroxyapatite at lower pressure and for a shorter duration tends to significantly modify the surface. In the case of the P2 samples, whose surface was modified at high pressure for 4 minutes, the Ra values are 6.18 μm, which is higher than the reference sample but lower than the P1 sample. This shows that applying hydroxyapatite at higher pressure and for a longer duration reduces the surface roughness compared to P1, with the layer being more uniformly distributed on the implant surface.

- The R sample shows surface irregularities along the baseline. The P1 sample has a slightly higher Rq than the reference sample. The P2 sample (whose surface was modified at high pressure for 4 minutes) has the lowest Rq value, reflecting the smoothest surface in this regard. This supports the Ra findings that higher pressure and longer duration lead to a more uniform surface.
- Thus, we can conclude that surface modification by air abrasion of hydroxyapatite has a clear impact on the morphology, wettability, and roughness of Ti6Al4V dental implant samples. The low-pressure treatment (P1) significantly increases roughness and decreases surface uniformity, while high-pressure treatment (P2) results in a less rough surface and a more homogeneous distribution of hydroxyapatite on the implant surface.

5.2. Own contributions and future research directions

The literature review and the experimental research conducted in this doctoral thesis laid the foundation for the development of original contributions, as summarized below:

In the theoretical synthesis section, the author made an original contribution by integrating the studied issue into a broader context of knowledge in medical engineering, but most importantly by identifying a practical method for modifying the surface of dental implants using equipment available in dental offices. The theoretical studies were conducted in such a way as to offer significant benefits both in terms of foundational knowledge and practical applicability, particularly:

- Contributions to the relevance of addressing the topic within global and national research on dental implants.
- Contributions to the creation of the experimental program and research methodology according to standardized methods, in compliance with EU requirements and unconventional approaches.
- Contribution to the study of the processes occurring at the implant-tissue interface.
- Contributions to establishing the theoretical and practical possibilities for the realization and modification of the characteristics of metallic biomaterials.
- Contributions to the modification of commercial dental implant surfaces. **Thus, the use of the air abrasion system for bioceramic coatings represents an innovative component and a practical solution.**

The research presented in this doctoral thesis does not exhaust the theoretical and experimental studies in the discussed field, but it brings original contributions:

- Two surface modification treatment variants were proposed, using the air abrasion system for hydroxyapatite coatings on Ti6Al4V alloy dental implants, resulting in two different types of experimental samples (P1, P2).
- Wettability of the surfaces was evaluated by determining the contact angle and surface free energy, with the development of a special working protocol to perform these determinations on different areas of the dental implants.
- Surface roughness was evaluated by determining the Ra, Rq, and Rz parameters, with the development of a special working protocol to perform these determinations on different areas of the dental implants.

Future research directions will focus on:

- ✓ Conducting studies to determine the mechanical resistance of the investigated implants;
- ✓ Studying the chemical by-products released from the degradation of the investigated materials to establish their potential effect on the human body;
- ✓ Investigating the biological properties by performing in vitro and in vivo biocompatibility tests.

Another future research direction with high potential is the optimization of abrasion parameters. It could be useful to explore other abrasion parameters (e.g., hydroxyapatite particle size, abrasion angle) to identify the optimal combination that would lead to better surface properties and improve the osseointegration process of dental implants.

Application of research findings

Articles (3 articles, including 1 ISI article and 2 BDI articles):

1. **Cristiana Ioana TATIA**, Maria IANCU, Alina ROBU, Octavian TRANTE, Iulian ANTONIAC, Anca Maria FRATILA, *Current trends in surface modification for dental implants*, European Journal of Materials Science and Engineering, Volume 9, Issue 4, 2024: 309-322, www.ejmse.ro, ISSN: 2537-4338, DOI: 10.36868/ejmse.2024.09.04.309, https://ejmse.ro/articles/09_04_05_EJMSE-24-246.pdf
2. **Cristiana Ioana TATIA**, Maria Mirabela IANCU, Aurora ANTONIAC, Alina NECȘULESCU, Alina ROBU, Marius Lucian VASILESCU, Iuliana CORNESCHI, Anca Maria FRATILA, *Comparative analysis of the surface properties of different dental implants*, U.P.B. Sci.Bull., Series B, Vol. 4, 2024
3. Maria Mirabela IANCU, **Cristiana Ioana TATIA**, Alina ROBU, Marius Lucian VASILESCU, Iulian ANTONIAC, Anca Maria FRATILA, *Current trends on materials and methods for teeth whitening*, European Journal of Materials Science and Engineering, Volume 9, Issue 4, 2024: 323-336, www.ejmse.ro, ISSN: 2537-4338, DOI: 10.36868/ejmse.2024.09.04.323, https://ejmse.ro/articles/09_04_06_EJMSE-24-247.pdf

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