euspen's 26th International Conference & Exhibition, Krakow, PL, June 2026
www.euspen.eu

Functionalization of tantalum oxide coatings on commercially pure titanium by PEO

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Abstract

The key point in biomaterials and implant research is biocompatibility. Metals and their alloys, such as titanium and tantalum, present favorable mechanical and physicochemical properties for both bulk applications and functionalized surfaces. Titanium is widely recognized for its use in the field of biomaterials, but tantalum has gained prominence in recent years. Titanium surfaces can be functionalized by depositing a tantalum layer, taking advantage of the positive properties of the bulk material combined with the benefits of a distinct coating, aiming for enhanced biological performance. This deposition is enabled by the technique of plasma electrolytic oxidation (PEO), and the analysis of this new surface is carried out using techniques such as profilometry and goniometry. Surface roughness was assessed as a physical parameter by profilometry, while surface wettability was assessed through the contact angle measured by goniometry. EDS analysis confirmed the incorporation of tantalum onto the surface, validating the proposed functionalization. This modification aims to replace titanium alloys that release cytotoxic ions, such as vanadium, with a biocompatible tantalum surface, representing a potential future option for biomedical applications.

Biocompatibility; surface functionalization; tantalum; biomaterials; implants; plasma oxidation

1. Introduction

Biocompatibility is fundamental to achieving success in biomedical implants [1]. The way a material creates its surface can favor cellular adhesion, improving integration with living tissues [2]. The interior of the material, or bulk, influences physicochemical properties that affect the human body's biological response [3].

Titanium, widely present in medical applications, is known for its good mechanical strength, biocompatibility, and its ability to form a stable passive layer that aids in osseointegration [4,5]. On the other hand, tantalum possesses a structure like trabecular bone [6] and promotes protein adsorption, optimizing the biological response. Applying a tantalum oxide coating over titanium can enhance properties such as surface energy, biocompatibility, and cellular adhesion. Despite the recognized biological potential of tantalum, there is still a scarcity of simple and reproducible deposition routes for incorporating Ta into titanium surfaces using PEO, with metrological control of roughness and wettability.

The Plasma Electrolytic Oxidation (PEO) technique offers the possibility of functionalizing titanium surfaces, promoting improvements in roughness and wettability [7,8]. Wettability, analyzed through goniometry, influences the cell-surface interaction and depends on topography and chemical composition [9]. Additionally, profilometry allows for the evaluation of morphological changes with high precision [10].

2. Methodology

Commercially pure titanium samples with dimensions of (25 x 40 x 1) mm were cleaned in an ultrasonic bath (L100 Ultrasonic Cleaner, Schuster, Santa Maria, Brazil) for 4 minutes using distilled water and neutral detergent (Extran MA 02, Merck KGaA, Darmstadt, Germany) for 15 minutes using a 2% (v/v). Subsequently, they were cleaned for the same duration in the same machine using only distilled water. This process was repeated a third time using isopropyl alcohol, followed by drying on filter paper.

For the deposition of the new tantalum layer via PEO, an electrolyte was prepared in which the samples were submerged inside the plasma reactor. The solution consisted of 0.5 L distilled water, tantalum oxide (Ta2OH5) at a concentration of 15 g/L, and potassium hydroxide (KOH) at 3 g/L. The mixture was prepared using a magnetic stirrer (Fusion Magnetic Stirrer, FUS-100, Corning Inc., New York, USA) under controlled conditions. A power supply (MKO Power Supply, Plasma Technics Ltd., Racine, USA) was used with the following pre-defined parameters: a voltage of 450 V, a frequency of 1000 Hz, duty cycle of 60%. The duty cycle corresponds to the fraction of time the voltage is applied relative to the total pulse cycle time, remaining active for 60% of the duration and inactive for the remaining 40%.

The exposure time across all samples varied from 10 to 30 minutes, aiming to visually achieve a uniform and standardized deposited layer for subsequent testing. The process resulted in the formation of a tantalum-containing oxide layer on the titanium surface, characterized by a rough appearance and a whitish color.

2.1 Profilometry Analysis

Surface roughness analysis was performed by profilometer (Dektak 150, Veeco Instruments Inc., Plainview, USA). A 'Standard Scan' type was employed, with a scan length of 2,000 µm and a duration of 60 seconds, totaling 20,000 data points per measurement. The scanning speed was set at 33.33 µm/s, moving from left to right. A stylus with a 12.5 µm radius was used, applied with a constant force of 3.0 mg. The vertical measurement range was 65.5 µm.

The following parameters were analyzed: arithmetic average roughness (Ra), root mean square roughness (Rq), and the surface asymmetry parameter (ASP). These analyses allowed for the precise observation of topographical variations resulting from the surface treatment applied to the samples.

2.2 Goniometry Analysis

The surface wettability of the samples was verified using a goniometer (Model 500 Advanced Goniometer, Ramé-hart Instrument Co., Succasunna, USA), equipped with fiber optic lighting and a manual microsyringe for droplet control. The analyses were performed using 2 µL droplets of distilled water and diiodomethane, deposited onto both the pure titanium surfaces and the tantalum-coated surfaces. Droplet images were captured by the goniometer's integrated system and analyzed via software to determine the contact angle between the droplets and the surfaces.

3. Results

In this section, the results obtained from the tests performed on commercially pure titanium samples and those functionalized with tantalum are presented. Initially, the wettability was analyzed using the goniometer.

Figure 1 illustrates how water interacts with different commercially pure titanium samples. With a contact angle of 102°, the first sample demonstrates a well-rounded droplet, indicating minimal water spreading and highlighting the characteristics of a highly hydrophobic surface [11]. Conversely, the other two samples show droplets with greater adsorption to the surface.

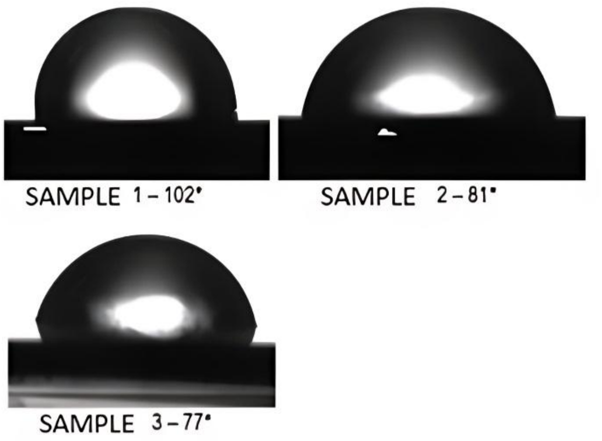

Figure 1. Goniometry assay of commercially pure titanium samples.

These variations in behavior between the liquid and the surface may be linked to subtle differences in the texture or treatment of the pure titanium surfaces, such as their polishing. In practical terms, surfaces that are rougher or possess lower surface energy in the outer layer tend to 'repel' water, causing the droplet to remain rounded as if resisting adhesion to the material.

These preliminary results underscore the importance of wettability in the success of implants. In subsequent tests involving tantalum oxide deposition, it will be possible to compare changes in wettability, which consequently affect the bioactivity and biocompatibility of the samples. A review of hydroxyapatite-doped coatings shows that typically more hydrophilic and rough/porous surfaces enhance interaction with biological fluids, cell adhesion, and proliferation, thereby improving biointegration [12].

Figure 2 shows the adsorption of the diiodomethane droplet by the functionalized surface sample. The way the droplet spreads demonstrates a certain degree of hydrophilicity in the material.

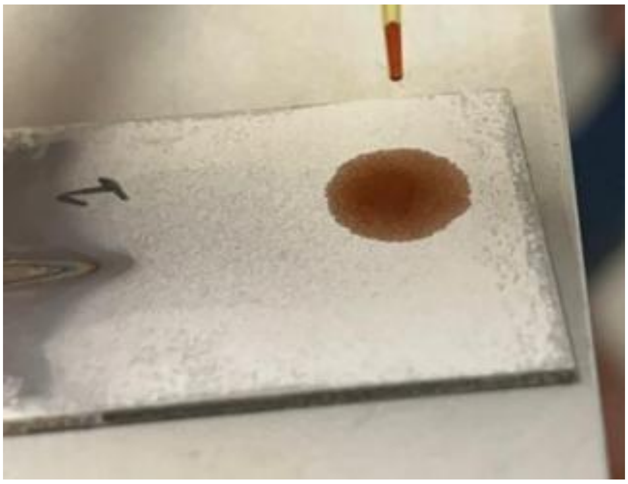

Figure 2. Goniometry assay with diiodomethane on tantalum oxide functionalized titanium samples.

Due to the rapid adsorption of the liquids onto the surface, analysis via contact angle becomes unfeasible. These results indicate that the deposition altered not only the surface characteristics but also the chemical composition of the material, optimizing its affinity for different types of fluids, which may impact cellular adhesion.

As shown in Figure 3, surface roughness (Ra) analysis was performed on twelve commercially pure titanium (cp-Ti) samples and twelve tantalum oxide-functionalized (Ti/Ta) samples using profilometry. The data demonstrate a substantial difference between the roughness values of the two surfaces. The Ti/Ta samples exhibit higher average roughness values, ranging between 10,225.7 nm and 16,239.1 nm, with the highest peak observed in sample 6. In contrast, the commercially pure titanium samples show significantly lower roughness, approximately three times less than the functionalized ones.

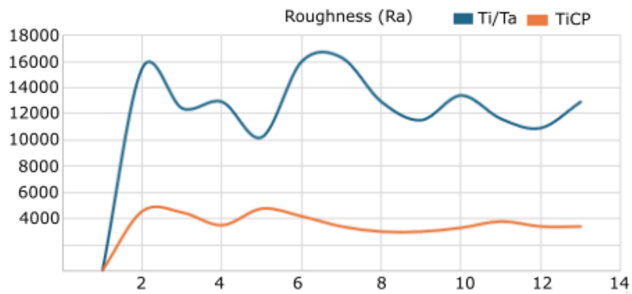

Figure 3. Surface roughness by profilometry of CP-Ti (commercially pure titanium) and Ti/Ta (titanium with deposition).

The increase in the roughness of the Ti/Ta samples indicates that the functionalization by tantalum oxide deposition via PEO contributed to the surface modification in a favorable manner. This increase in roughness is related to a larger contact area with the liquid, which potentially enables cell adhesion and osseointegration in biomedical applications. As shown in Figure 4, EDS reveal the predominant presence of Tantalum on the sample surface at two different points. Furthermore, the porous aspect of the surface morphology is verified. The micrograph of the deposited layer shown in Figure 5 reveals a heterogeneous and irregular surface, which is a common characteristic of coatings obtained by Plasma Electrolytic Oxidation (PEO). The points labeled as 'Spectrum' indicate regions selected for EDS chemical analysis, chosen to represent different morphological features of the same surface. Selecting different points allows for the evaluation of deposition uniformity and the identification of possible local variations in chemical composition. Surface cavities can host cells from body fluids that would come into contact with the surface, favoring cell adhesion and biocompatibility. Nano/micro topography directly influences cell adhesion [13]. As the increase in roughness was confirmed by profilometry, it can be stated that the functionalized layer possesses biological potential.

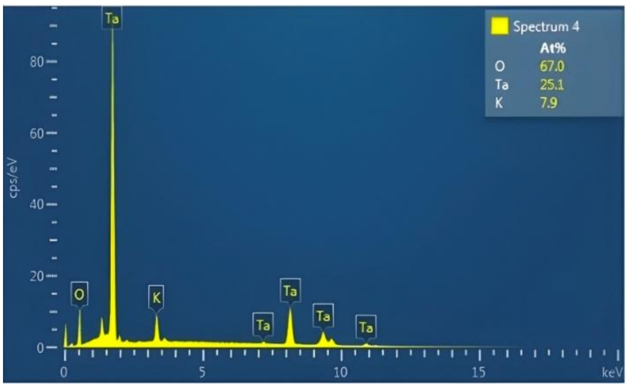

Figure 4. Chemical composition obtained by EDS of the titanium surface functionalized with tantalum oxide, at point 4. Source: The author.

4. Discussion

In this work, an increase in the wettability and surface roughness of the Ti/Ta alloy is demonstrated; these factors are associated in the literature with a better biological response, although they have not yet been directly evaluated by biological assays in this study [1].

Several studies demonstrate that the wettability and roughness of metallic or polymeric surfaces directly modulate protein adsorption, cell adhesion, immune response, and, ultimately, biocompatibility [1,14,15,16,17,18].

The results achieved in this research prove that the functionalization of the titanium surface with tantalum oxide via PEO promotes significant changes in surface properties. The goniometry test demonstrates an increase in wettability, highlighted by the decrease in the contact angle with water and diiodomethane. Profilometry revealed an increase in the average roughness of the functionalized samples, emphasizing a topography favorable to cell adhesion. SEM and EDS analysis confirmed the presence of Tantalum in the functionalized layer.

These alterations contribute to biocompatibility and osseointegration, reinforcing the efficacy of the PEO technique with tantalum oxide deposition for biomedical applications. Future studies may further the investigation with wear resistance and corrosion analysis.

The PEO process in a Ta-containing electrolyte produced a Ta oxide layer with a porous morphology and high roughness, confirmed by EDS and profilometry. Results demonstrated a significant increase in roughness and a drastic shift in wettability. The surface exhibited rapid droplet adsorption, rendering traditional contact angle measurements unfeasible. This behavior indicates a strong affinity for biological fluids, characteristic of superhydrophilic surfaces.

5. Conclusions and future work

Since both variables (roughness and chemical composition) change simultaneously, it is not possible with the current data, to quantitatively separate the individual contribution of each to the increase in wettability. Nonetheless, the presence of Ta is confirmed by EDS and the increase in roughness by profilometry, indicating a combined effect.

These findings suggest that the proposed PEO treatment is an effective and relatively simple method for Ti/Ta surface functionalization. Furthermore, the ability to incorporate tantalum while controlling surface topography presents a promising alternative to conventional Ti-alloys that release cytotoxic ions, such as vanadium, thereby enhancing the biocompatibility of orthopedic implants.

This study validates PEO-based Ti/Ta functionalization as a robust method to enhance biocompatibility through improved wettability. The results provide a clear proof of concept, with tantalum incorporation and topography control strictly verified by EDS, SEM, profilometry, and goniometry.

The research is ongoing. Future corrosion assays and in vivo tests will be conducted to verify and confirm the biocompatibility capacity of the functionalized topology.

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