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Deposition of Ta2O5 Film on Commercial Pure Titanium Disk by Modified Reactive Plasma Sputtering Technique

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Abstract

Background: Now days the interest of implantology is to improve Osseo integration by find materials which accelerate bone formation at bone implant interface to provide immediate or early loading after placement of dental implant and eliminate waiting period which is un favorite and disturbs patients. Titanium is most common material for dental implant, but still need some modification for surface properties chemically and physically by using an other material such as tantalum which is gaining more attention as a new metallic biomaterial. Coating the surface of implant is an important way for development of surface properties of titanium. plasma Ta2O5 used for surface modification, which has several advantages such as increasing surface roughness, changing surface topography and increasing the wettability of the surface.

Aim of study: To evaluate the action of Ta2O5 coating by modified plasma sputtering technique of commercially pure titanium disk on surface roughness, wettability and surface chemical composition in comparison to non-coated surface.

Materials and methods: All tested group were two groups in this study which include un -coated commercial pure titanium disks and Ta2O5 coated commercial pure titanium. Modified reactive plasma sputtering technique was used to coat CpTi with Ta2O5 at 4,6,8, hr. Analysis of surface characterization by x-ray diffraction (XRD) analysis, scanning electron microscope (SEM), energy dispersive spectroscopy(EDS) were carried out on the coated surfaces of the disks, contact angle measurement was achieved by applying drop of saline on the surface of coated disks and compared to non- coated one.

Results: The result of Ta2O5 coated specimen at times (4,6,8) showed that 8 hr. coating time was the best time. X-ray diffraction analysis show new peak formation for CpTi coated witht Ta2O5 disk which was not present in non- coated CpTi disk. The results showed that the rough surface was more and better distribution in CpTi coated with Ta2O5 disk than non- coated CpTi disk which improve by electron scanning microscope, wettability of Ta2O5 coated disk was more than wettability of non- coated CpTi disk.

Keywords: *Ta2O5 film, commercial pure titanium disk, modified reactive plasma sputtering technique*

Introduction

Surface improvement of dental implant become much advanced than it was earlier [1]. Titanium is material of choice for dental implant which shows a favorable combination of intrinsic properties for the fabrication of dental implants such as low modulus of elasticity, low specific weight, high strength to weight ratio, very high corrosion resistance, easy surface coating and excellent general biocompatibility [2]. Acceleration of the Osseo integration of Cp Ti results in reducing the non-functional time period of the implant, increase its applicability in alveolar bone with low quality, minimize the failure rates, and cause minimum discomfort for patient. [3]. Increasing roughness and change surface properties physically and chemically is one of the methods used in increasing and accelerating the Osseo integration process. Surface coating can be used for changing surface topography, increasing surface roughness and in increasing the wettability of the surface. [4] Chemically and physically reactive plasma discharges are widely

used to modify the surface properties of materials. [5]. Tantalum (Ta) is stable chemical element can stably exist in the surface layers of the substrate materials. Ta is one of the promising materials in used in dental fields [6]. The Ta components offer a low modulus of elasticity, high surface frictional characteristics, and excellent Osseo integration properties (i.e. Bioactivity and biocompatibility) [7] The stable Ta2O5 protective film can provide better bioactive property and corrosion resistance than that of TiO film. [8]. Surface-coated implants are reported to have less failure and can support heavier and more dynamic forces. Several clinical studies have analyzed the success and failure of implants in diabetic patients. [9]

Material and Method

Sample preparation

Disks of Titanium were prepared (5×2mm) diameter and thickness respectively which were cut from Cp Ti rod using Bench Nibbling machine (TAURUS 7000-W6 CNC, Italy). The specimens were abraded by using Si C grinding paper with different grits started from 80 grit, and continued by 120, 230, 400, 600, 800 and 1000 grit to get free scratch and flat surface. All disks were polished to obtain a mirror polished and smooth surface. After that all specimens cleaned by ultrasonic cleaner with ethanol 99.8%. was carried out.

Surface coating by Ta2O5

By using modify Dc glow discharge reactive plasma sputtering system, the sputtering technique was performed. Number of the total samples were 10 disk which divided as 1 un coated CpTi disk and 9 coated CpTi disk with Ta2O5 (3 disk for each time). Then the coated disks sub divided according to the times of coating (three times 4,6 and 8 hr.) into 3 groups. The cause of 3 disk coated in same time was to send each sample to specific test for time consuming.

The sputtering procedure started with placing the clean and polished samples on the center of base of anode electrode. evacuating process of chamber to high vacuum (≈1x10^{-}5mbar) using high vacuum system consist of rotary and turbomolecular vacuum pumps to ensure the complete removing of the heavy gases like hydrocarbons. Power supply (negatively charged voltage 3.5 kV). For sputtering process, the voltage was gradually applied using variac until the required energy achieved (applied voltage and current), The pressure was 2x10^{-}2 to 7x10^{-}2mbar and this pressure was achieved by feeding the bombardment and reactive gases. The appropriate voltage and amber were adjusted precisely by regulator until intended sputtering glow (purple color, which standardized for each gas) is achieved. One of the most important modification done to convert normal plasma system to sputtering plasma system was replacement of electrode position, so the cathode placed in upper part and anode electrode placed in lower part of chamber. A Target (cathode) and anode disk of stainless steel. The cathode faced the anode with 7 cm distance between them, which provides electric field for the gas to be discharged. The electrical electrodes and the associated dc-power supply of 5kV.The bottom of the stainless steel disk cathode electrode are covered with tantalum sheet which regarded as target. The clean and polished samples were placed on the anode in the center of base which regarded as substrate. The cathode was connected to the D.C. power supply, while the grounded chamber served as anode. All the samples were cleaned by argon plasma sputtering for 15 min prior to the Ta2O5 coating process by applying a bios dc voltage of 100 Von the anode. For reactive plasma sputtering procedure, the following process parameters were used: argon was used as bombardment gas and oxygen as reactive gas. The process was carried out at various sputtering times (namely 4, 6 and 8 hours). The reactive gas oxygen was introduced into the evacuated chamber and the flow rate was adjusted until the pressure was stabilized at the desired pressure (1×10^{-}2 mbar), then the argon gas introduced to the chamber until the sputtering pressure of 5×10^{-}2 mbar achieved. After the sputtering process completed, the samples were kept until ambient temperature in the vacuum chamber was reached.

Phase Analysis by using X-Ray Diffraction

Surface analysis for Phase's distribution was performed for non-coated CpTi, CpTi coated with Ta2O5 it has been done by using X-ray Diffraction Facilities (SHIMADZu 6000, Japan) using Cu Kα radiation. XRD analysis were performed at room temperature in the 2θ range from 30˚- 80˚ with a 0.05˚ step and counting time of 5 secs per step. The indexing of the data and the diffraction peaks were identified according to the powder diffraction files. (PDF), received from ICDD (Intimations' Center for Diffraction Data).

Scanning electron microscope examination

All tested group were examined by using SEM

(JEOL-JSM-5600) for examinations of the changes occurs on the coated layer of Ta2O5 during the plasma sputtering technique. Samples for SEM were prepared as cross section and then mounted in double face metal tape (electrically conductive) to make the sample holder electrically conductive before entering the samples into the SEM Chamber. VEGA3 TESCAN, SEM HV: 20 KV, SEM MAG:7.50kx, VIEW FIELD:27.7

energy dispersive spectroscopy analysis

chemical structures and relative concentrations for non-coated CpTi and coated CpTi with Ta2O5 discs were assessed via energy dispersive spectroscopy (EDS). EDS use of the X-ray spectrum radiate a solid sample with a focused beam of electrons to obtain a localized chemical analysis. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle. Qualitative analysis involves the identification of the elements in the spectrum and is fairly straightforward owing to the simplicity of X-ray spectra. Quantitative analysis determination of the concentrations of the elements. Finally, element distribution images or 'maps' can be produced.

Atomic force microscopy examination

Atomic force microscope able to detect both conductive and nonconductive surfaces on the atomic scale. The AFM relied on the scanning technique and provided a high-resolution 3D image from the surface of the sample. A sharp tip at the end of the cantilever is in contact with the surface of the development and the sample displaced with piezoelectric scanners. The force on the tip causes deflection to measure with tunneling capacitive or optical detectors such as interferometer laser in this technique, the standard pressure applied to the joint is zero (to prevent any surface deformation).

Wettability test

Non-coated CpTi and Ta2O5 coated CpTi (grade 2) disk 10×5 mm diameter and thickness respectively were used by dropping equal amounts of normal saline (0.25ml) from graduated container on each disk, non-coated CpTi and coated with Ta2O5 for (4,6,8 hr.) and measure the angle formed between titanium disc surface and drop of normal saline. The best wettability achieved for the sample was selected for the (8hr.) measurement done after 20 second from putting the drop on disk surface, and digital camera about 15 cm from disk. The images were analyzed using the program Image Tool version 5.02 for Microsoft Windows. [10]

Result

Surface characterization by using X-Ray Diffraction.

A: Before Heat treatment:

XRD patterns of the control CpTi specimen and Ta2O5 coated by modified reactive plasma sputtering after different sputtering time (namely 4, 6 and 8 hours) are demonstrated in Figure (1). and these data were indexed according to the Powder Diffraction Files (PDF) for the hexagonal αTi (JCPDS-ICDD file # 44-1294), Ta2O5 (JCPDS-ICDD file # 25-0922, 33-1391and 33-1390), and Ta2O5 (JCPDS-ICDD file # 34-0977). The diffraction peaks of the uncoated CpTi were found to be corresponding to (100), (002), (101), (102), (110), (200), (112) and (201) α-Ti at 2θ values 35.00°, 38.30°, 40.05°, 52.9°, 62.8° ,70.6°, 76.25°and 77.35° respectively. The patterns of the plasma sputtered specimen for 4 and 6 hours show wide peak in the range of 2θ 20-30o which seems to be due to the formation of not fully crystallized Ta2O5, whereas the pattern of the specimen sputtered for 8 hours show clear and prominent Ta2O5 peaks indicating the formation of Ta2O5 corresponding to the reflections 001, 200 and 1117 at 2θ 22.90, 28.79 and 49.12.Also its very clear there are a shift in the 2θ position of the CpTi reflections towards the higher 2θ indicting the change in titanium crystal cell volume.

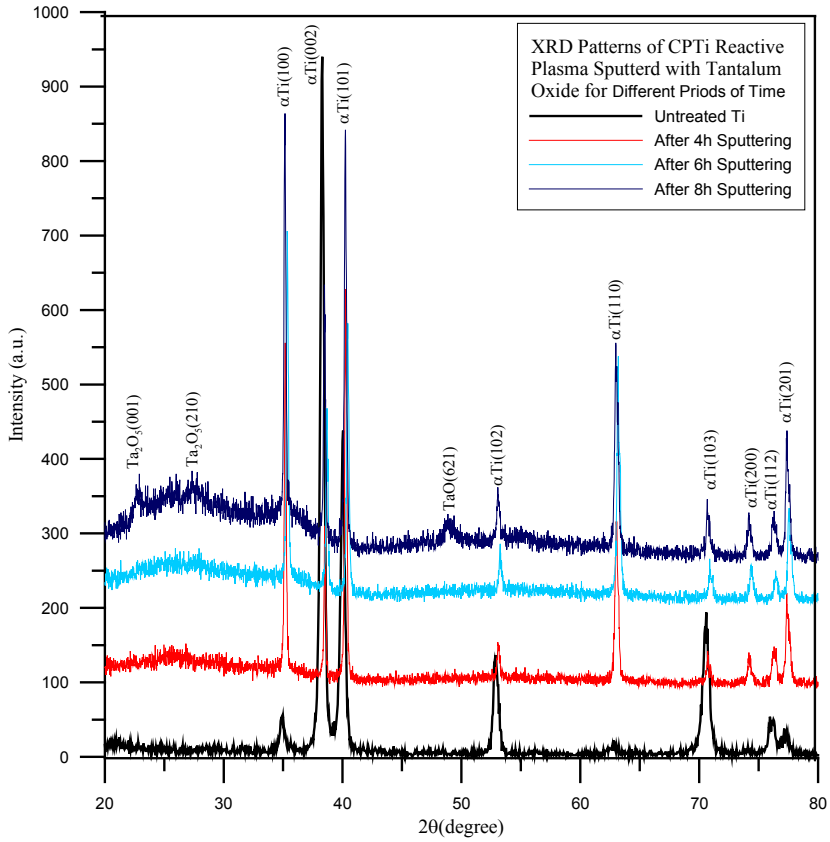


Fig (1): XRD patterns for uncoated CpTi and coated specimen with Ta2O5 at (4, 6, and 8 hr.)

B: After Heat treatment:

The CpTi specimen's coated with Ta2O5 for sputtering time 8 hours before and after heat treatment are shown in Figures (2). The pattern of the heat coated specimen show prominent new peaks indicating the formation crystalline tantalum pentoxide Ta2O5. The peaks are assigned to 001, 200, 270, 211, 3100, 092, 3111, and 3171 at 2θo 23.06, 28.79, 32.88, 37.12, 49.62, 50.98, 56.24 and 64.4 respectively. Also it's noticed that only one reflection belongs the tantalum monoxide Ta2O5 which are 621 at 2θo 52.52.

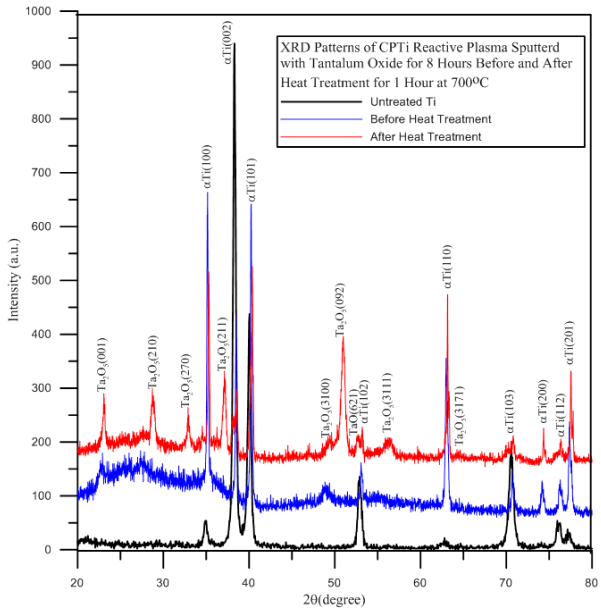


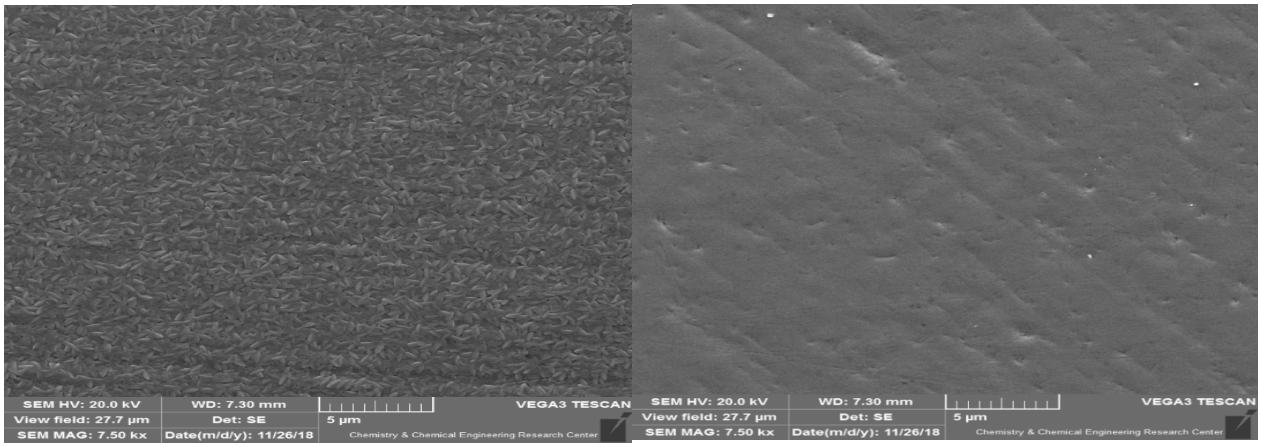
Fig (2): XRD patterns of uncoated Cp Ti and coated with Ta2O5 for 8 hours before and after heat treatment.

Surface characterization using Wettability test

From the result of wettability test, 8 hr. coated disk with Ta2O5 give more hydrophilic surface. Contact angle of fluid drop in plasma sputtering with Ta2O5 was less than non-coated disk. Un coated CpTi disk with contact angle 60◦ , Ta2O5 disk coated for 4hr with contact angle 65◦ , Ta2O5 disk coated for 6hr with contact angle 45◦ and Ta2O5 disk coated for 8hr with low contact angle 30◦.

Surface characterization using SEM

A: Topographic

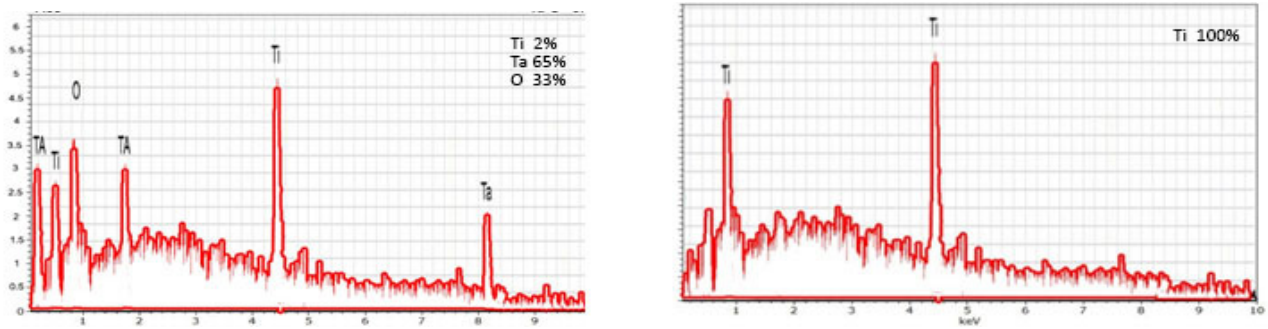


The scanning electron microscopy images of CpTi disk before coating in (figure 4a) revealed a relatively flat and smooth surface. While the surface morphology of CpTi plasma sputtering samples with Ta2O5 for 8 h showed a fully arranged nanochips and uniformly distributed in (figure 4b).

A: CpTi disk B: CpTi disk coated with Ta2O5

Fig (4): SEM.A/CpTi disk without coating B/CpTi disk coated with Ta2O5 for 8 hr.

B: chemical composition by EDS



chemical structures and elemental composition were assessed for all experimental groups which include CpTi and CpTi coated with Ta2O5. Regarding the chemical structure analysis for CpTi figure (5a) show tow titanium peaks with these value (kα 4.512 and lα 0.452) which resemble alpha phase of titanium. For second group of disk coated with Ta2O5, there were two peaks of titanium which resemble alpha phase, two tantalum peaks and one peak of oxygen with following value (kα 4.512 and lα 0.452) (lα 8.146 and mα 1.712) (kα 0.525) respectively as shown in figure(5b). Elemental composition of tested group as follow in (fig a) titanium was 100%, in (fig b). percentage of titanium was 2%, tantalum was 65% and oxygen was 33%.

A: CpTi disk B: CpTi disk coated with Ta2O5

Fig (5): EDS.A/ CpTi disk without coating B/CpTi disk coated with Ta2O5 for 8 hr.

Discussion

The new trend in implant research is to increase and accelerate the osseointegration of Cp Ti which may result in decrease the non-functional time period of the implant, increase its applicability in alveolar bone with little quality, cause minimum discomfort to patient, and minimize the failure rates. [11] One of the methods used in increasing and accelerating the osseointegration process is to modify the surface properties of the implant [12]. Chemically and physically reactive plasma is widely used to modify the surface properties of materials. [13] In order to improve surface properties of CpTi, tantalum is a material with specific properties which is used in this research as a coat material with oxygen gas to develop Ta2O5 surface on CpTi disk by modified sputtering technique which has several advantages such as changing surface topography, increasing surface roughness, and in increasing the wettability of the surface. [14]. Reactive plasma coating constitutes a simple, dry technique, which does not harm the environment, of low cost and does not comprise the intrinsic properties of the biomaterial, affecting only its surface. [15]

Financial Disclosure: There is no financial disclosure.

Conflict of Interest: None to declare.

Ethical Clearance: All experimental protocols were approved under the College of dentistry. University of Karbala, Iraq and all experiments were carried out in accordance with approved guidelines.

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