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The effect of different surface roughness on the Ti6Al4V alloy surface implant by ceramic composite materials coated

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Abstract
In this research was study the effect of roughness types on the surface of substrate implants. The Ti6Al4V alloy was prepare by Dip coating method with solution include (HAp (a hydroxyapatite has 2 um particle size with purity is 5N (99.999%)), TiO_2 (a Titania pure rutile phase has 2um particle size with purity is 4N (99.99%)) and polymer PVB)) after mechanical and chemical treatments with heat treatment at 500^{o}C. In order to increasing biocompatibility surface alloy. Atomic Force microscopy measure the value roughness of surface alloy Ti6Al4V (43.8 nm for mechanical and (23.4nm) chemical treatments. The microstructure of samples were identify by using Scanning Electron Microscopic SEM and Optical microscope OP analysis. The efficiency biocompatibility of the coated surface were seen with biomimetic tests In vitro studies were carried out in simulated body fluid with HP 7.4. OP and SEM shows presence the elements of coated layer Hap (which appearance of many forms such as dendritic, lumbar and sheets shapes) and TiO2. The intensity result of EDS shows the concentration of Ca, P and Ti or mechanical treatments is more than in chemical treatments. Also initial TiO_2 and especially HAp surface on the sample was found to have suitable an inter grain to growth the apatite in a solution SBF. The increasing in the concentrations of Ca and P after immersion in SBF was show EDS Also the components of SBF appearing in other peaks (C,O,Na,K,Cl and Mg) that is mean the surfaces is biocompatibility and this confirm by Optical microscopy, according to the result the coating will be compatible with human tissue. The value of V Hardness samples was (120, 90, 83) Kg/mm for Ti6Al4V alloy (uncoated, Coating (chemical treatment) and Coating (mechanical treatment)) respectively, shows are decreasing and more approached to the human bone value compared with uncoated of Ti alloy.

Keywords: Oxide titanium; Hydroxyapatite; Dip coating method; Mechanical and chemical treatments.

1. Introduction
The basic role in successful of biocompatibility and integration of an implant is the surface which is direct contact with the adjacent tissues. There is now the good understanding that design of the novel tissue-engineering medical and the life time of orthopedic implants productions may be associated with local cell-material interaction and control biological tissue restraint. Implant material specifications such as topography, roughness, pours and implant surface composition can effects on the cell response to implant material and implant –bone interfaces [1, 2]. It's important to gathering the properties of materials nature with high corrosion resistance, wear resistance, bioactivity, and biocompatibility, mechanical properties to

manufacturing artificial implants with developed mechanical, physical, and chemical properties. There are many way to the solution of enhance the biocompatibility of the surface properties and the problem and the results of load-bearing prosthesis such as mechanical properties is lead to the deposition of many multifunctional coatings with different types biomaterials on their working parts. HAp hydroxyapatite is the must ceramic material and significantly used in biomedical application due to special good chemical, physics and biocompatibility [3,4]. Ti alloy also use in the application of dental and orthopedic implants because excellent properties such as biomaterial behavior, high wear, corrosion resistance and hardness. The material composition coating and the roughness of surface implant was always effect on the cell attachment adhesion, proliferation and differentiation. Attachment between implant and surface tissue is usually abound on rough surfaces more than the smooth ones [5,6].The mechanical methods(apply external action by the application of physical forces such as machining, polishing) and chemical methods(are used acid and alkaline etching) are more used to prepare and obtain specific either a rough or a smooth surface topographies are subtraction and also removal of contamination. [7,8]. A basic of bioactive material use for implant is that induces a specific biological response at the interface of the material that results in the formation of a bond between the tissues and the implant materials. A common characteristic of all bioactive implants is the formation of a hydroxyapatite (HAp) layer on their surface when implanted in the human body. The HAp phase have chemical characteristic is equivalent in structure and composition to the mineral phase of bone ,so it is use is widely used in biomedical application. To improve the mechanical strength of layer coating surface , composite methods have been developed such as ceramics composite(such as HAp,TiO2,Zr,ZnO,Al2O3..) with different way of coating [9,10]. Titania TiO2 is also used in biomedical application because its phase and biocompatibility and enters into installation of bone. The presence porosity in the surface is very important condition for successful implant because a large amount of pores is introduced into the materials, reaction kinetics and inter-growth processes can be enhanced. A high bioactivity material, HAp is coated on the surface of the substrate in this way the hard tissue can be in contact with large surface area which has porosity due to ceramic coated layer and that is be more bioactive .The hard tissue or bone is growth well induced due to high porosity level and high bioactive surface [11,12].The aim of the present study the effect of roughness created with chemical and mechanical methods on the surface of metal coated with composite ceramic(HAp and TiO2) material by comparative analysis of cell adhesion in vitro tests.

2. Materials and experimental techniques

The material was used HAp (a hydroxyapatite has 2um particle size with purity is 5N (99.999%)) and TiO_2 (a Titania pure rutile phase has 2um particle size with purity is 4N (99.99%)) (provided from VTFM (Vacuum Thin Film Materials).The titanium alloys (Ti-6Al-4V) use as substrate alloy was GR2 ASTM F136 (Baoji Jinsheng Metal Material Co. Ltd). According to the manufacturer.

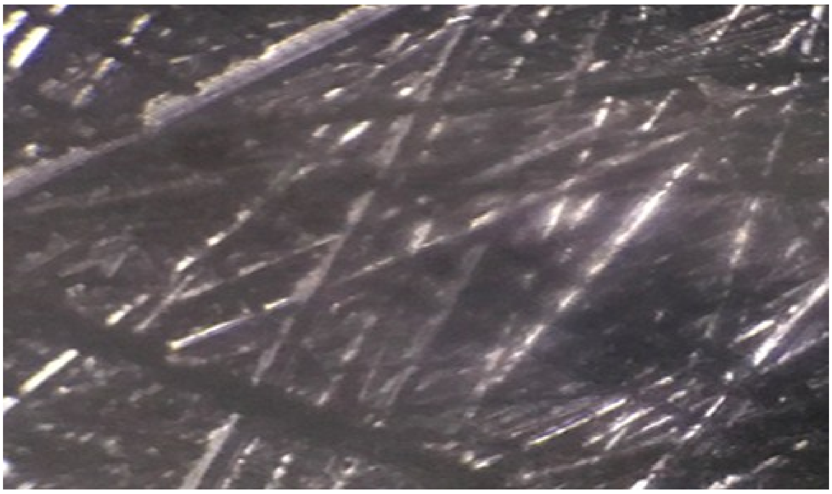
The work includes the use ceramic composite of TiO_2 and HAp were deposited on substrate of Ti-6Al-4V alloy using spin coating method after modification surface by mechanical and chemical methods to change the roughness surface.The value of roughness was determined by atomic force microscopy AFM and topography of surface was determined by optical microscopy OPTwith a magnification of 100 μm.

2.1. Surface modification

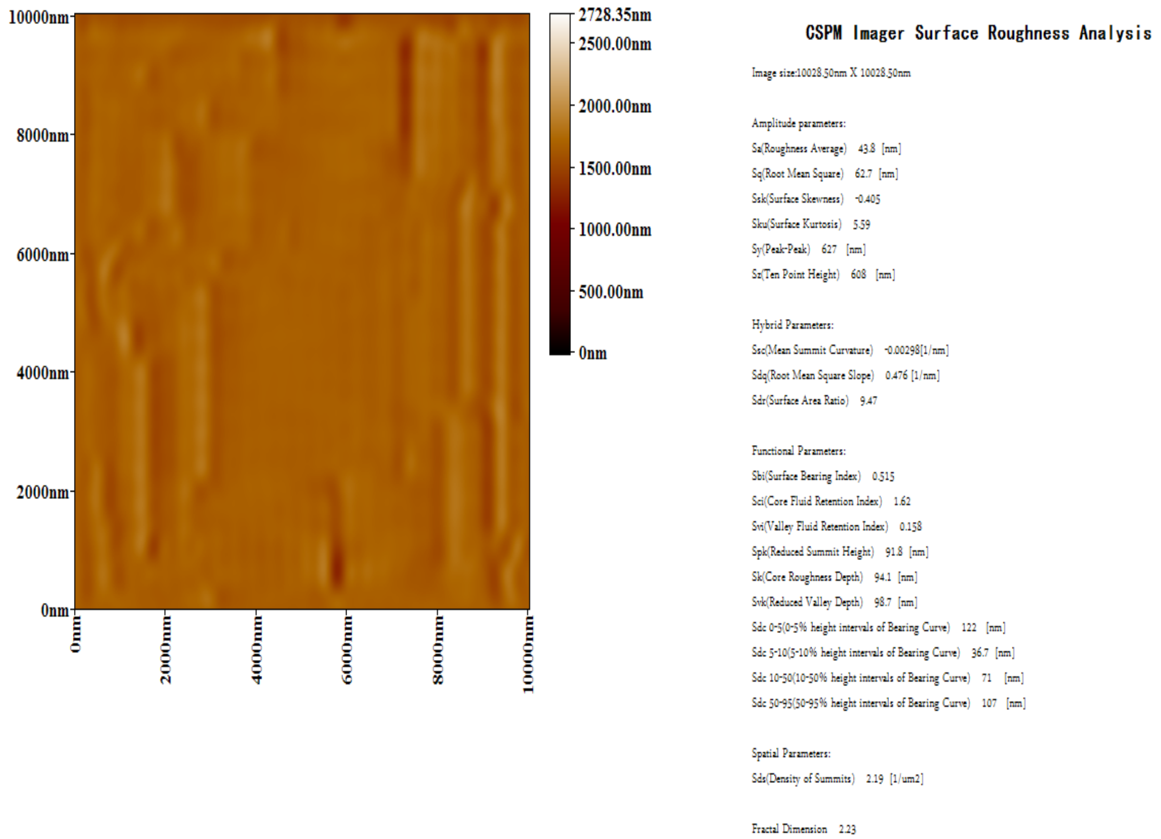
a. mechanical surface modification method for metal alloy was grinding and polishing by using SiC paper with different grit range size (800,900, 1000,1200 ,1500 and 1800 μm) . The value of roughness (43.8 nm), as shown in Figure1.
b. The chemical methods modification was include etching the metal alloy immersion in the solution (HF:10g, HNO3:30g and deionized water:60g) for 3min. The value of roughness (23.2 nm), as shown in Figure 2.
c. All the samples washed out the strips by an ultrasonic using distilled water and ethanol bath within 20 seconds respectively at room temperature.
d. Composite powder from TiO2(12.g)+HAp(13.g) was mixing by using electrical blender for 5 min. the last composite TiO2+HAp(25 g) was precursor with polymer PVB with chemical form is ($C_6H_{14}O_2$) (0.35 g/L)(based on the zhitomirsky) use to prepare the appropriate solution, milted in 1000ml of CH2CH3OH with continuous stirring by using magnetic stellar for 2hrs,[13].
e. The substrates (after mechanical and chemical modifications) was coated by immersed vertically in the chemical samples in the solution mentioned above using dip coating method. within 1.5 min

we immerse the 1st sample, the second sample is immersed twice in the solution (first immersion within 1.5 min then let it to dry then we immerse it again for 1.5min) the results are samples with multilayers, let it to dry again finally the dip coating method conditions must be equal in velocity and time at each time of immersion and withdrawal [14].

f. Finally after the strips dried and appropriate deposition of the thickness with thick film (HAP & TiO_2), all the samples was heat treatment at 500°C for 1 h by using furnace.

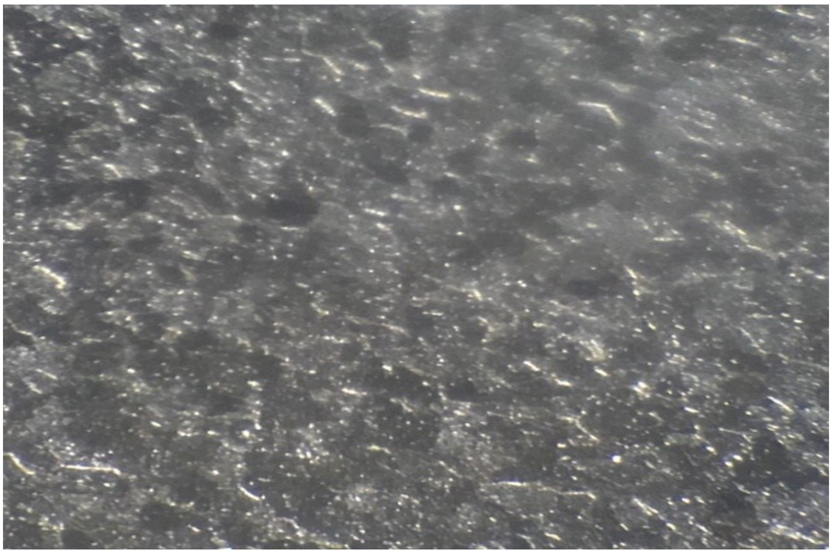


(a)

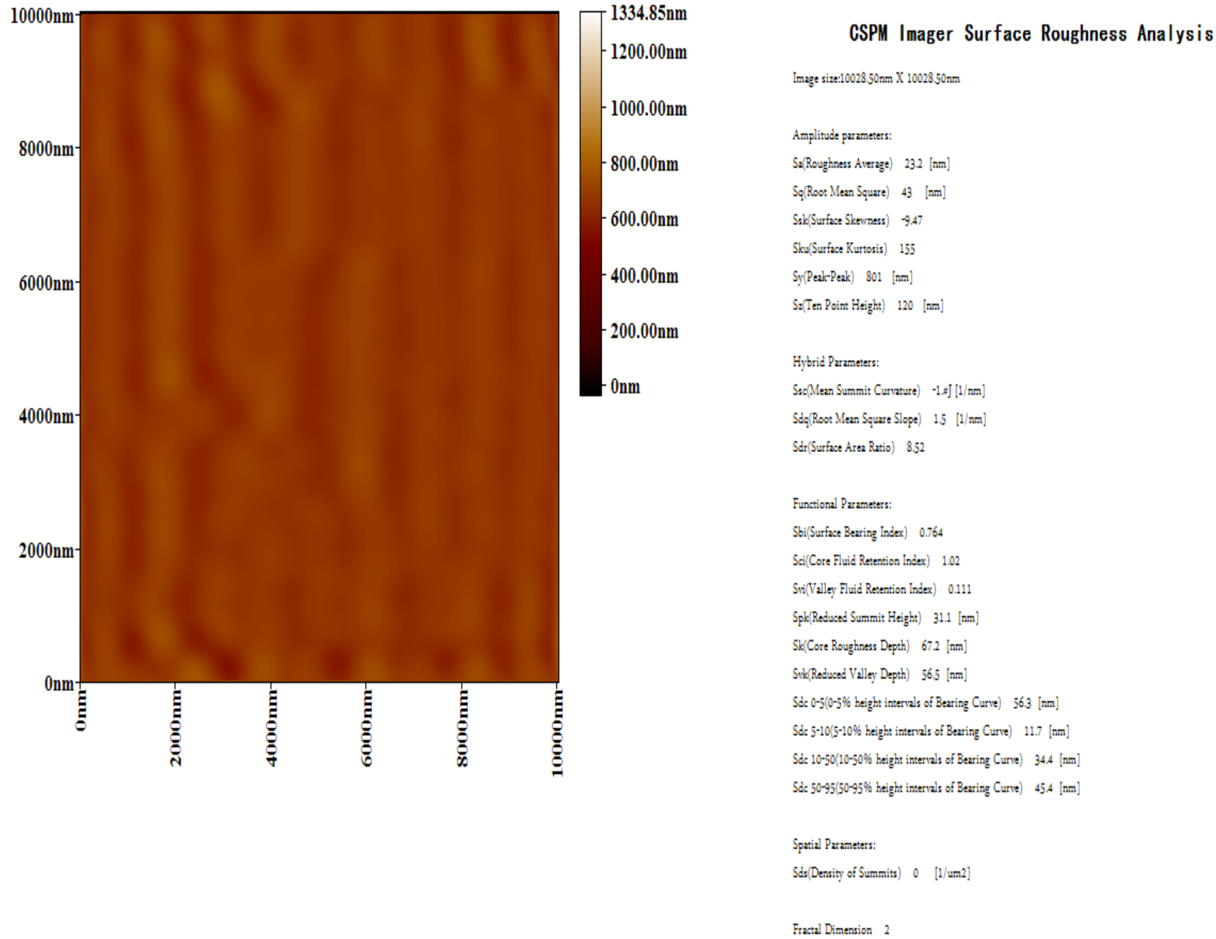


(b)

Figure 1. The topography and roughness of (Ti-6Al-4V) after mechanical modification; (a) OPT, (b) AFM.



(a)



(b)

Figure 2. The topography and roughness of (Ti-6Al-4V) after chemical modification; (a) OPT, (b) AFM.

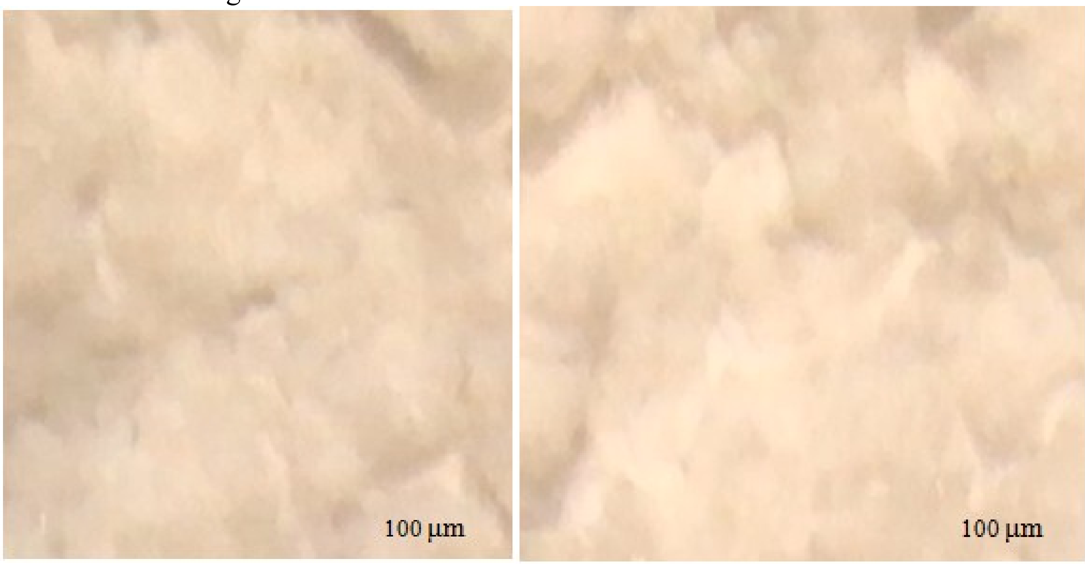
2.2. Biocompatibility experiments

The biocompatibility experiment include immersing all the samples with the multilayered thick film, in simulated bode fluid(SBF)a pH of 7.4 and 37 °C, for a three weeks [15].

3. Results and discussion

3.1. Optical microscopy

Figure 3 shows the image of optical microscopy with a magnification of 100 µm for substrate Ti-6Al-4V alloy after coated with thick film (HAP & TiO2) and heat treatment 500°C for Mechanical and Chemical treatment. The whit colure film full coated substrates with nonhomogeneous distribution is due to the method used in the coating.

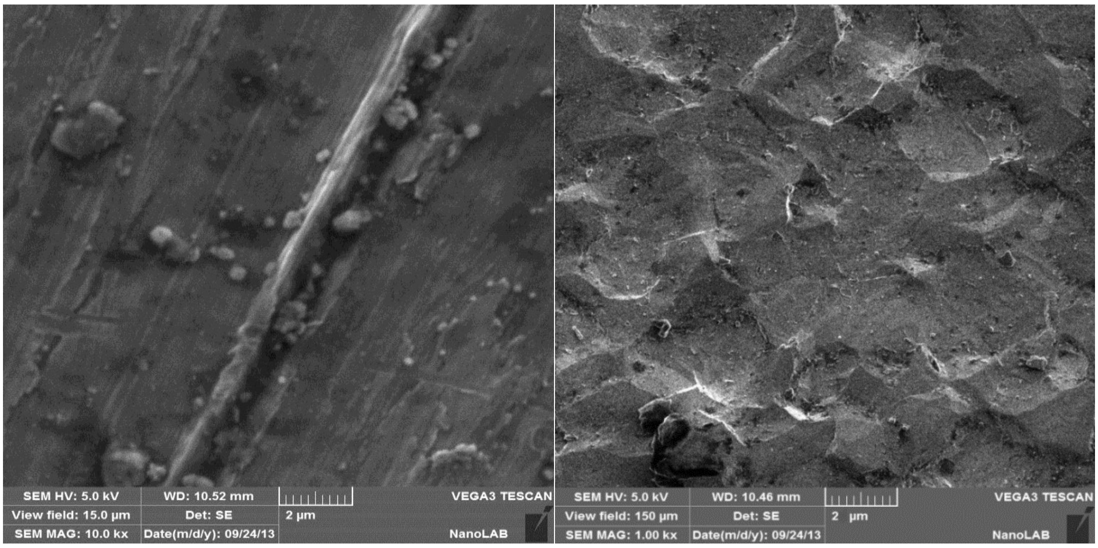


(a) (b)

Figure 3. Optical Microscopy for substrate Ti6Al4V alloy coated with thick film (HAP + TiO_2) with magnification of 100 µm after treatments; a: Mechanical treatments, b: Chemical.

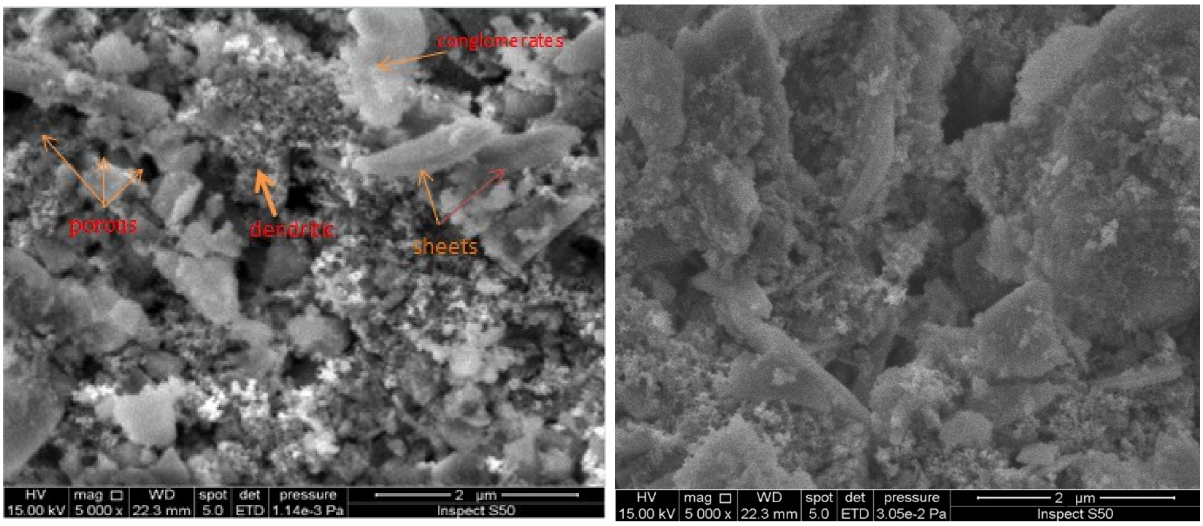
3.2. Scan Electron Microscopy (SEM) examination

Figure 4 (a, b) top view SEM image microstructure surface of Ti6Al4V with Mechanical and Chemical treatment respectively. The morphology of surface shows a grooves and lines in mechanical and look like concavity in chemical treatment, that is agreement with results of OP in Fig. 1&2.Figure 5 (a,b) shows conglomerates are components of coating materials due to thick coating. The dendritic and sheets shapes belong to HAp and the spherical shapes belong to TiO_2 particles [16]. The coated are covered fully the surface interface with titanium substrates and have porous with no crack .The vacancies and porous found on the implant surface of samples may have good positive effect on implant and tissue bio integration. Such as, osteoblasts (bone forming cells) [17].



(a) (b)

Figure 4. Top view SEM image microstructure surface of Ti6Al4V alloy with (a) Mechanical and (b) Chemical treatment.

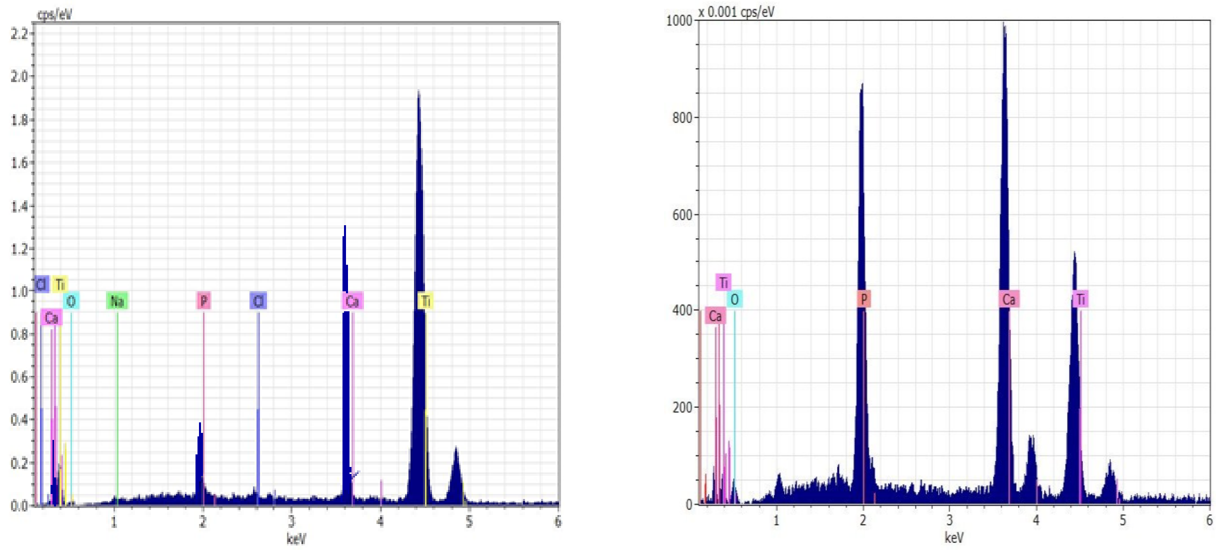


(a) (b)

Figure 5. Top view SEM image microstructure surface of Ti6Al4V alloy coated with thick film (HAP + TiO_2) a: Mechanical treatments, b: Chemical.

3.3. EDS energy dispersive X-Ray spectroscopy

the chemical composition of the Ti-6Al-4V alloy after coated with (TiO_2+HAp) and the chemical properties elemental was investigate by using energy dispersive X-Ray spectroscopy (EDS) is carried out in Fig. 6(a,b) . The deposition of Ca and P on samples are belong to HAp, the energy transitions elements Ca Kα and Lα are(3.69 and 0.34)KeV respectively , the energy transition for P Kα at energy of 2.01 KeV. The energy of 4.93KeV which are for TiKβ, belong to TiO2 coated layers and substrate Ti-6Al-4V alloy. Also the elements appearing in other peaks (C and O), the energy transitions elements O Kα is 0.5KeV. The elements of C belong to component of polymer .The concentration of Ca, P and Ti in EDS results of fig.6 for mechanical treatments is more than in chemical treatments. Appearing the elements Ca ,P,O and Ti which are the components of coated materials are agreements with results of OP and SEM in Figs.(4 and 5) respectively .



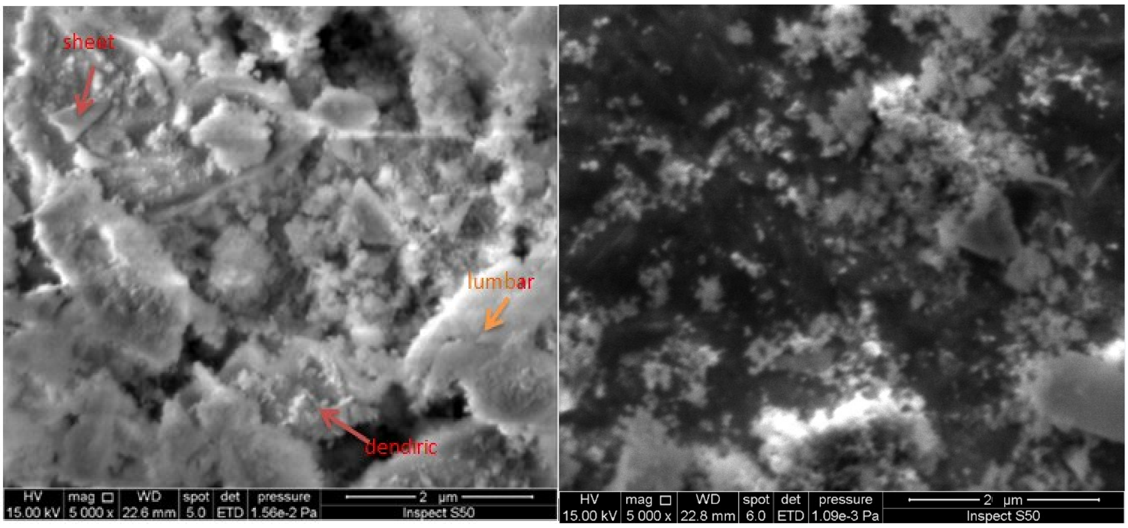
(a) (b)

Figure 6. EDS spectra concentration for Ti-6Al-4V alloy coated with thick film (HAP & TiO_2) (a) Mechanical and (b) Chemical treatment**.**

3.4. In vitro Tests coating morphology of thick film (HAP+TiO2)

3.4.1. Scan Electron Microscopy (SEM) examination

Figure 7. (a, b) top view SEM image microstructure surface of Ti6Al4V alloy for mechanical and chemical treatments coated with thick film (HAP + TiO2) and after immersion in SBF. The increasing in thick coated layer is clear for two samples after immersion compared with Fig.6. (a,b). The appearance of many forms for HAp dendritic, lumbar and sheets shapes

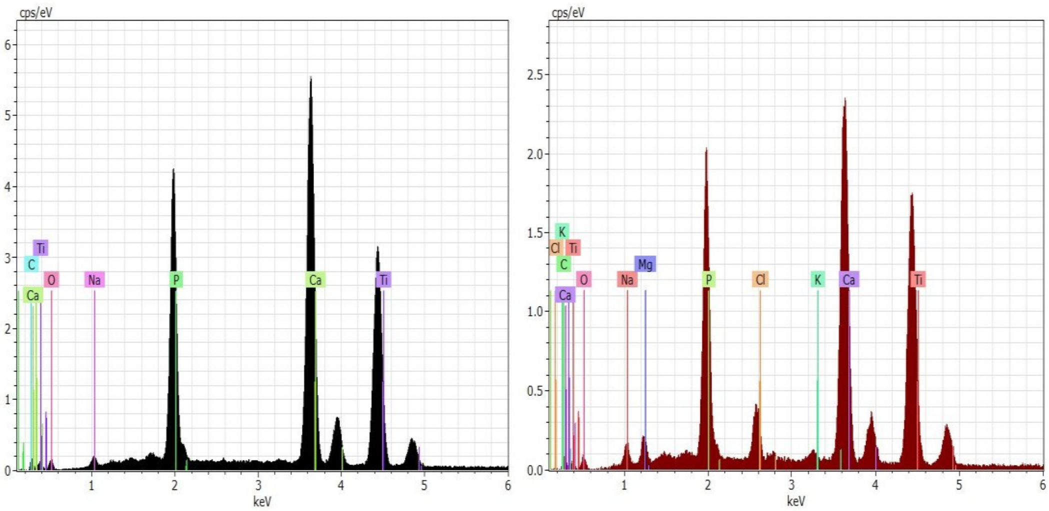


(a) (b)

Figure 7. Top view SEM image microstructure surface of Ti6Al4V alloy coated with thick film (HAP + TiO_2) after immersion in SBF a: Mechanical treatments, b: Chemical.

3.4.2. EDS energy dispersive X-Ray spectroscopy (EDS)

In order to investigate the chemical composition and compassion of the Ti-6Al-4V alloy after coated and immersion in SBF, the chemical properties elemental analysis energy dispersive X-Ray spectroscopy (EDS) is carried out in fig. 8(a,b) .The deposition of Ca and P on samples are belong to HAp, the energy transitions elements Ca Kα and Lαare(3.69 ,0.34)KeV respectively , the energy transition for P Kα at energy of 2.01 KeV. Also the components of SBF appearing in other peaks (C,O,Na,K,Cl and Mg), The energy transitions elements O Kα is 0.5KeV, energy transition for Na Kα is 1.041KeV,energy transition for Cl Kβ is 2.81 KeV. Energy 1.25 KeV for Mg Kα and (0.86 ,3.3) KeV for K Kα, and 4.93KeV which are forTiKβ , belong to TiO2 coated layers and substrate Ti-6Al-4V alloy. There are increasing in the concentrations of Ca and P compared to in fig 6. (a,b), initial HAp surface on the sample was found to have suitable an inter grain to growth the apatite in a solution SBF.



(a) (b)

Figure 8. EDS spectra concentration for Ti-6Al-4V alloy coated with thick film (HAP & TiO2) after immersion in SBF (a) Mechanical and (b) Chemical treatment.

3.5. Mechanical testing V Hardness

Load applied force=0.49N for 10 sec on the surface of Ti6Al4V alloy uncoated and coated with thick film (HAP & TiO_2) after (a)Mechanical and (b) Chemical treatment for 30 seconds were taken at 5 different places on the surfaces and \average of these values were considered in this result. From the results of V Hardness were shown in Fig (9) and Table (1) the value of V Hardness for the surface of samples decreasing and more approached to the human bone value compared with uncoated of Ti alloy.

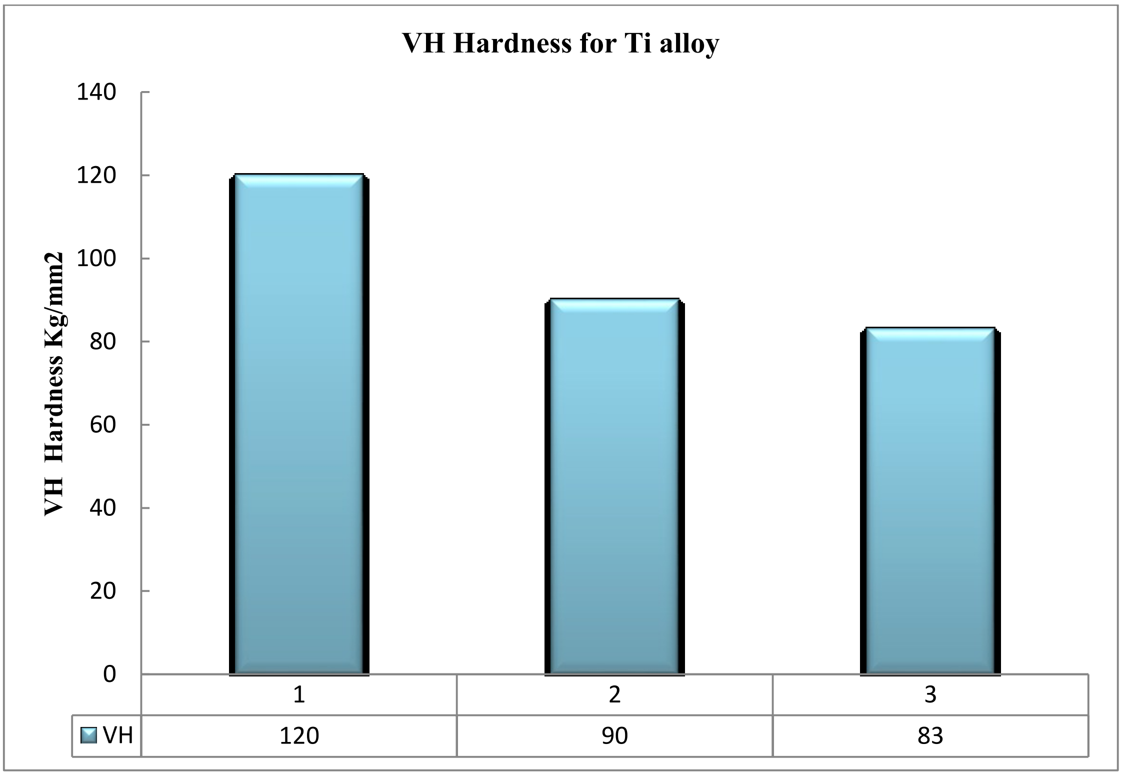


Figure9. V Hardness for samples after and before coated.

Table (1): The value of VH Hardness for samples.

No	Types of samples		VH Hardness Kg/mm2
1	Ti6Al4V alloy uncoated		120
2	Coating with (HAP & TiO2) thick film	(chemical treatment)	90
3	Coating with (HAP & TiO2) thick film	(mechanical treatment)	83

4. Conclusions

1- Atomic Force microscopy measure the value roughness of surface alloy Ti6Al4V (43.8 nm for mechanical and (23.4nm) chemical treatments.

1. Dip coating method was successful for full coated substrate Ti alloy with ceramic (TiO2+HAp) for mechanical and chemical treatments.

2. The Optical and SEM shows presence the elements of coated layer Hap (which appearance of many forms such as dendritic, lumbar and sheets shapes) and TiO_2.
3. The intensity result of **EDS** shows the concentration of Ca, P and Ti or mechanical treatments is more than in chemical treatments witch agreement with result of OP and SEM.
4. In vivo test, there are increasing in the concentrations of Ca and P compared to concentration after immersion in SBF. Also the components of SBF appearing in other peaks (C,O,Na,K,Cl and Mg)
5. The value of V Hardness for the surface of samples was (120,90,83)Kg/mm for Ti6Al4V alloy (uncoated , Coating (chemical treatment) and Coating (mechanical treatment))respectively , shows are decreasing and more approached to the human bone value compared with uncoated of Ti ally.
6. Also initial $TiO_2$and especially HAp surface on the sample was found to have suitable an inter grain to growth the apatite in a solution SBF.

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