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Investigation the Coating of Hydroxyapatite on Titanium Substrate by Pulse Laser Deposition

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Abstract:

This study investigated and prepared a coating for surgical implants by using hydroxyapatite (HA) empowers characteristic bone that developed at a medium for prosthetic the parts of human body. HA is the generally manufactured from both Calcium (Ca) and Phosphate (P) to produce ($Ca_{10}(PO_4)_6(OH)_2$) that used as a base material for covering mineral embeds because of its incredible biocompatibility and comparable the synthesis and structure to sclerous tissues of the human body. HA, coatings on titanium substrates have been produced by Pulses laser deposition (PLD) techniques. HA used in this search pressed at pressure (150MPa) with particle size (2.745 µm) and used as a target in coating by (PLD) techniques. Surface characterization studies of the coatings such as XRD, SEM, AFM and EDX to detect the amount of (Ca) and (P) in coating layer were carried out. Then test the micro-hardness, surface roughness for HA coating .Corrosion behavior for uncoated and coated samples with various number of pulses in Hank's solution by using OCP and the potentially static polarization test were achieved also, , in this test we obtained a greatly improved in corrosion resistance of the samples B1 after coating by 99.88%.

Keywords: Coating, Hydroxyapatite, Prosthetic, Biocompatibility, Tissues and Pulses laser deposition.

1.1 Introduction

Metal implants were first introduced by Lane in 1895 since he found metal plate for bone break obsession. In the early progress, metal implants had several issues including corrosion and insufficient strength problems [1]. Metallurgical materials, for example, treated Steels, pure1 Titanium and Titanium composites and Cobalt combinations are ordinarily utilized as bone embeds because of the great quality of these minerals, sturdiness and generally low corrosion rate in vivo, [2]. The biocompatibility of embed metals is of extensive concern since they can consume in the antagonistic body condition [3]. Prior research has demonstrated that these issues can be overwhelmed by saving bioactive hydroxyapatite (HAP) (Ca10 (PO4)6(OH) 2) coatings on metal implants [4]. Covering of bioactive materials utilizing hydroxyapatite (HAp) in the metallic embed has numerous preferences, including enhanced erosion opposition of embed surface and upgraded biointeraction with the encompassing tissues [5].

Bio-active materials is a term used to refer to the materials that will be active inside the human body and it will be interact with the bones around it or with the soft tissue in some cases depending on Bioactivity Index (IB) which introduced by (Hench) as shown in below to measure the level of bio-activity :

$$I_B = 100/ t\ 0.5bb \qquad \ldots. (1)$$

Whereas: t 0.5bb = the time required to get bonding interface for more than 50%. In addition, this usually occurs during the time depending on kinetic modification of the surface, generated by implantation of the bio-active material inside the live bone. The interaction of ion exchange between the biologically active plant and the surrounding fluids' body which consequences in the development of a biologically active carbonate apatite layer on the chemically equivalent and crystalline agar of the bone mineral age. Prominent examples of biologically active substances are synthetic hydroxyapatite (HA), and bioglass [6].

Hydroxyapatite (HA), usually written as Ca10 (PO4)6(OH) 2 is a member of the apatite group of ceramics. In addition, apatite terms refer to deceit or deception and the main origin for this term is the Greek word (a pate) and the cause of this name return to the variation of the shape and colour [7]. The (HA) is used as an artificial bone because it has the same chemical structure as the natural bone in spite of devoid it from organic elements like collagen and polysaccharides. Also, HA has very good biocompatibility and bioactivity, so many medical engineers are interested in employing HA in their work applications [8], [9]. HA is synthesized and used to production different implants forms (solid and permeable) or coating other implants [10].

Wang, et.al., (2009) They deposited both Hydroxyapatite (HA) and fluoridated Hydroxyapatite (FHA) as a coating on titanium substrates by depending on electrochemical method. Where they use many different (F-) ions concentrations were incorporated into the apatite structure as a result from the adding of NaF to the electrolyte. The results showed that coatings properties were uniformly dense and have a very thin coating layer with (~ 5 μm) thickness, [11].

Xuhui ZHAO, et.al. (2009). Hydroxyapatite coatings were prepared directly on titanium's anodized by electrostatic deposition technique in a simulated modified fluids of body. The composition, structure and effectiveness of the coating were examined by using a scanning electron microscope (SEM), XDR diffraction analyser by X-ray and Fourier infrared spectroscopy (FTIR) methods. The results showed that the homogeneous and pure hydroxyapatite could be gained without any treatment after coating, [12].

Liu et al. (2011) examined the deposition of hydroxyapatite on titanium, st.st 316, copper and aluminum. A novel seeded hydrothermal deposition method was used after forming a nanoscale layer of HA by electrochemical process. The effect of reaction heat, solution pH, calcium/ phosphorus ratio and deposition time on the HA were investigated. The developed implants for this study are promising for bioactive applications because it allows promoting bone growth, [13].

S.Jafari et .al. (2011) used sol-gel deposition method to investigate the difference between coated St.St.316L and Ti-6A1-4V alloy from mechanical and bioactive point of view. Micro hardness and bonding strength of substrates were examined. In addition, the samples were subject to SEM, XRD, EDS, AFM and optical microscopy to show their performance. The results showed that the surface roughness of HA coated on Ti-6A1-4V was noticed more than the case with 316L, [14].

Barry et .al. (2014) investigated and compared the properties of the hydroxyapatite coated on several types of metals using both atmospheric plasma spray (APS) and Co-Blast techniques for deposition. Three types of substrates were used (e.g. titanium, cobalt chromium and St.St.316). Changing phase of implants (HA and substrates) was noticed when APS was used. On the other hand, no phase altering with Co-Blast method employed for deposition [15].

2. Experimental part:

2.1 Pulsed Laser Deposition Technique

Pulsed-Laser Deposition (PLD) is a physical vapor deposition (PVD) method that has gained approval for the high quality growing of multicomponent oxide thin films, [16]. The output of a short laser pulse (10 to 30 ns) is focused onto a solid target. The laser rapidly raises the surface temperature of a small portion of the target well beyond the vaporization and is collected on a nearby substrate. The consistent non-steadiness evaporation of multicomponent materials and the transfer of the target configuration to the deposited film make accessible high-quality thin films of materials that cannot be placed by other PVD methods, [17], as shown in figure(1).

In this work, a plate of titanium was cutting into 3 samples each one which have dimensions (4.5*2.5*0.1) mm. Then these samples were rinsed in distilled water. Then these samples degreased with acetone and ultrasonically cleaned (for 10 minutes using acetone as a medium), and ethanol, respectively using ultra sonic cleaning device.

Preparation of HA target include, a 15 g of nano powder was mixed with (3ml) of poly vinyl alcohol (PVA) as a binding material. Then, the mixture was mold in (20 mm) diameter by compressing mould, and pressed at pressure (150 MPa) using compacted device, to get target as shown in Figure (2). The mould was pre-lubricated to reduce friction and to release the compact model easily. After that, the target was dried using the dry box at 150℃ for 4 hrs for dehumidify and PVA releasing.

2.2 Deposition Procedure and Parameters

To accomplish the work objectives described in section (2.1), first, the samples of titanium coated with HA according to achieve the plan as shown in Table (1). HA deposition was done using the parameters shown in Table (2).

2.3 Tests:

In the research, the following tests were performed to evaluate the performance of HA coating layer on Ti substrate:

2.3.1. Light Optical Microscope (LOM)

Involved identification and measurement of the phase's shape and grain size are some characteristics of grain boundaries. Each of these has distinct characteristics. The microstructure evaluated with (600-x) magnification using Olympus microscope manufactured by Japan.

2.3.2. AFM Analyzer

Atomic force microscopy (AFM, contact mode, spm AA3000 Angstrom advanced Inc., USA) was used to observe the morphology (Roughness, depth morphology of film) in (college of Material engineering /University of Babylon).

2.3.3. SEM Analyzer

In present work SEM is used to reveal the microstructure of both uncoated and coated samples. It has been used to characterize the morphology and microstructure of the coating surface in terms of uniformity, the test has been done at the (University of Technology / part of Production and Metallurgy).

2.3.4 EDX Analyzer

Energy Dispersive X-ray (EDX-7000) in University of Technology/part of Production and Metallurgy was used to test the chemical synthesis of sample surface.

2.3.5. Hardness Test

Vickers Hardness (TH-717 Digital Micro Vickers Hardness Tester) was measured the hardness of HA films, at load (300g) and holding time 15 seconds in (college of Material engineering/University of Babylon).

2.3.6. Thickness Test

The thickness measuring device was used to measure the thickness of HA films on Ti substrates, in (Babylon/College of Material Engineering/University of Babylon).

2.3.7. Surface roughness test

The surface roughness of a HA coated Ti sample was measured by using the (TR-100 surface roughness tester), which is located at the University of Babylon, Faculty of Materials Engineering. The device passes on the sample surface to measure the surface roughness. The device has a sensor that records the roughness of the sample surface and takes the reading directly from the device screen. The accuracy of the device (± mμ0.01).

2.3.8. Electrochemical Test

The corrosive behavior of titanium studied in Hank's solution. The chemical analysis for Hank's solution is illustrated in Table (3) and pH of it at 37C° was 7.4.

2.3.8.1 Open Circuit Potential (OCP)

The experimental arrangement for the measurement of open circuit potential is described. A 500 ml capacity glass electrolytic cell is used. The tests were performed with the specimen submerged in a Hanks solution the potential current of electrode is calculated in comparison with a Saturated Calomel electrode (SCE).

2.3.8.2 Potentiostatic Polarization

Electrochemical experiments were performed in three electrode cell containing and electrolytes Hank's solution which chemical composition is shown in table (3), [18].

The test was performed by gradual increasing in the potential by a scanning ratio 0.4 mV/s, that starting from 450 mV as an initial potential below the open circuit potential and the scanning continued up to 450 mV over the open circuit potential.

Corrosion rate measurement is got by applying the following equation [19].

Corrosion rate (mpy) = $0.13 i_{corr.}(E.W.)/A.\rho$ …(2)

Where:

E. W. = equivalent weight (g/ eq)

A = area (cm^2)

ρ =density (g/ cm^3)

0.13 = metric and time conversion factor.

$i_{corr.}$ = current density ($\mu A / cm^2$).

The improvement percentage is calculated for coated samples, using the following equation [19]:

Improvement percentage =$(CR^0 - CR / CR^0)^* 100$ …(3)

Where:

CR° =the corrosion rate of uncoated sample (without coated).

CR =the corrosion rate of coated sample (with HA coated).

3. Results and discussion:

3.1. Light Optical Microscope (LOM)

The microstructure of HA coating on titanium plate with number of laser pulses (4000, 6000, and 8000) using the magnification of the microscope (600x) and (200x) are shown in figures (3) and (4).

3.2. AFM Results

Figures (5) show results from AFM of HA coating deposited at different pulses laser. It can be noticed that deposition rate of HA particles is affected by increasing the pulses laser to (8000). It can be seen that the roughness decreased with the pulses increasing to (8000).

3.3. SEM Results

Figure (6) shows SEM micrographs of 150MPa HA samples deposited at different pulses and at 300˚C substrate temprature. It is clear from the figure increasing the pulses results in improvement of the HA Films. The particles of HA are deposited in order to produce clusters which looks like a heavy a aggregated composition on the base material. Furthermore, more pulses will be beneficial in improving the film growing, density and microstructure.

3.4. EDX Results

The percentages of (Ca), (O) and (P) of HA films and for (Ti) for the samples (B1, B2, B3) are shown in figures (7-9) respectively. As the number of pulses, increase the ratio of (Ca) and (P) increases in the coating layer. This is expected due to the increased amount of HA precipitation as the pulse increases. This is same as El-Sayed et al, investigation al, [20].

3.5. Hardness Results

The effect of laser pulses number on the HA coating hardness are shown in figure (10), the significant effect of pulses number on the resulted hardness can be observed .Hardness of uncoated Samples are improved after coating it with HA. Furthermore increasing of pulses from 4000 to 6000 to 8000 could improve the hardness from (247 HV) to (252 HV) to (277 HV), this results are agreement with P. Rajesh et.al, [21], Such improvement is due to the improvement in depth morphology, distribution and increasing in HA thickness which can be clearly observed in AFM results, as in figure (5), and SEM results as in figure (6). Most likely the pulse increasing could implant more HA particles on the substrate surfaces.

3.5. Thickness Results

For B1 sample the thickness is 2.2 µm, the thickness increased to 3.2 µm for B2 sample, and then the thickness reach to 4.15 µm for B3 sample. Anyhow with increasing number of laser pulses, the deposition rate increased and the thickness increased due to increase the deposition layer also as shown in Table (4).

3.7. Roughness Test

The roughness of HA film for B1sample is 0.482 µm, and it increased until reached 0.510 µm for B2 sample, and then the roughness reach to it is high value 0.965 µm for B3 sample. The effect of laser pulses number on the surface roughness of HA coating is showing in figure (11).

3.8. Corrosion Tests

3.8.1. Open Circuit Potential (OCP)

Time Measurements

The OCP - time was measured with respect to SCE in Hank's solution at 37°C for uncoated samples and coated samples (HA Coating).

3.8.2. Potentiostatic Polarization

This test was done by using the Potentiostatic polarization test in Hank's solution for uncoated and HA coated samples at 37 °C, with 1cm2 surface area.

Corrosion parameters (corrosion potential, corrosion current, and corrosion rate), extracted from these curves, are shown in Table (5)

The data listed in Table (5) show that attrition parameters at temperature (37°C) both uncoated samples and coated samples imbedded in solution of Hank. So, the polarization curves are shown in figures (12–15). The corrosion potential of all coated samples shows a significant shift to a positive direction and have more noble potential compared to uncoated sample.

From the Table, it is clear that (B1, B2,and B3) samples have current densities and corrosion rate much lower than current densities and corrosion rate of uncoated sample, which indicate that HA coating acts as a barrier against attack of aggressive an ions, affectively improve the corrosion resistance of Ti implant, [22],[23].

From Table (5) It's clear, that the samples showed the corrosion resistance of Ti sample is increased after coating with HA comparison with Ti uncoated sample, [24].

Corrosion ratio for the implant inside lives' bodies can be reduced by coating the sampling with HA, due to the reduction in iron releasing from the metal surface. Where the HA has a favorite characteristics which lead to applied it widely in different implants protection. One of the implantation process requirements for prostheses, there should be a contact between the tissue around the bones and the metal prosthesis. Where the availability of HA in metal coating for the implant will lead to produce the bonding rapidly between the tissue around the bones and the metal prosthesis. Many reasons lead to apply the HA in coating the implant because HA is a biocompatible material so the implants have both strength and biocompatible at the same time which lead to induce the ingrowths of bone and tissue surrounding it as well as the chemical reactions that produce bonding. Also, the availability of HA in implants metal coating will increase the resistance of the metal against corrosion in condition of

soaking the metals in biotic solutions by strengthen the chemical bonds and decrease the metal iron releasing [24],[25].

Conclusion:

Based on the obtained results, the following conclusions are made:

1. The films hardness increased form (178 HV) for uncoated sample to (277 HV) for coated sample
2. The thickness of HA film increased with increasing the number of pulses from (2.2 μm) for B1sample to 4.14 (μm) for B3 sample.
3. Increasing number of pulses is more beneficial in films growing and distribution.
4. In Hank's solution however, the improvement in corrosion resistance was (99.88%).
5. Another interesting increasing in biocompatibility was achieved during the work for the coated sample compare to the uncoated Ti sample.

Tables:

Table (1): Coating Plan

Target pressure MPa	Substrate temp.℃	Quantity of samples
150	300	3

Table (2): HA Deposition Parameter.

Parameter	Available ranges	Selected value
Power	**1-1000 mJ**	**1000 mJ**
Frequency	**1-6 Hz**	**6 Hz**
Pulses	**1-15000**	**4000-6000-8000**
Laser wave length	**1064 nm**	**1064nm**
Pulse duration	**10 nsec**	**10 nsec**
Vacuum pressure	**10-5 mbar**	**10-5 mbar**

Table (3) samples coding

Sample code	Number of pulses
---	**Uncoated**
B1	**4000**
B2	**6000**
B3	**8000**

Table (4): Chemical Composition of Hank's Solution [10]

NO.	CONSTITUENT	(mg/l)
1	NaCl	8.0
2	$CaCl_2$	0.14
3	KCl	0.4
4	$NaHCO_3$	0.35
5	Glucose	1.0
6	Mg $Cl_2.6H_2O$	0.1
7	$Na_2HPO_4.\ 2H_2O$	0.06
8	$KH_2\ PO_4$	0.06
9	Mg $SO_4.\ 7\ H_2O$	0.06

Table (5): The Thickness of Layer HAP Coating with Various pulses

Number of pulses	Thickness (μm)
B1	2.2
B2	3.2
B3	4.15

Table (6): Illustrate the Corrosion Potential (Ecorr.), Corrosion Current (icorr.), Corrosion Rate (CR) and Improvement Percentage of coated Samples in Hank's Solution

Sample code	Icorr. µA/cm2	Ecorr. mV	Corrosion Rate (mpy)	Improvement Percentage%
A	13.94	-702.7	9.6252	-
B1	1.47	-691.3	0.0109	99.88
B2	1.34	-715.4	0.0115	99.87
B3	4.84	-708.1	0.0418	99.56

Figures:

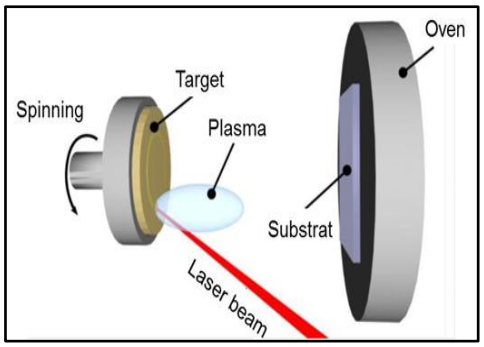


Figure (1): (A) Schematic of the PLD Process,[9].

Figure (2): One of Targets after Pressing Process

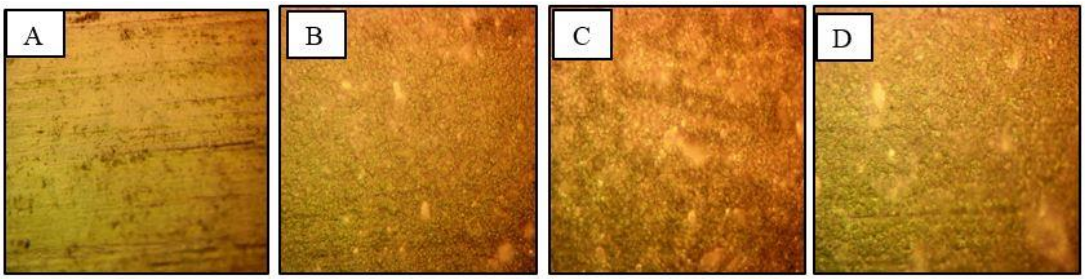


Figure (3): Microstructures of HA Coating (A) A Sample (B) B1 Sample (C) B2 Sample (D) B3 Sample

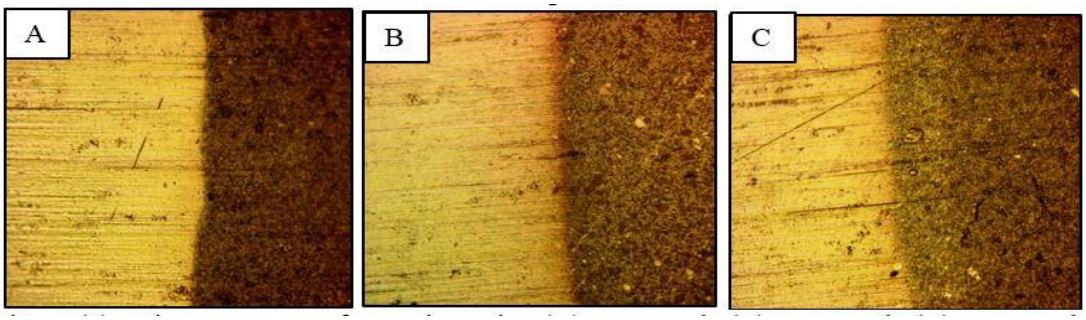


Figure (4): Microstructures of HA-Ti Coating (A) B1 Sample (B) B2 Sample (C) B3 Sample

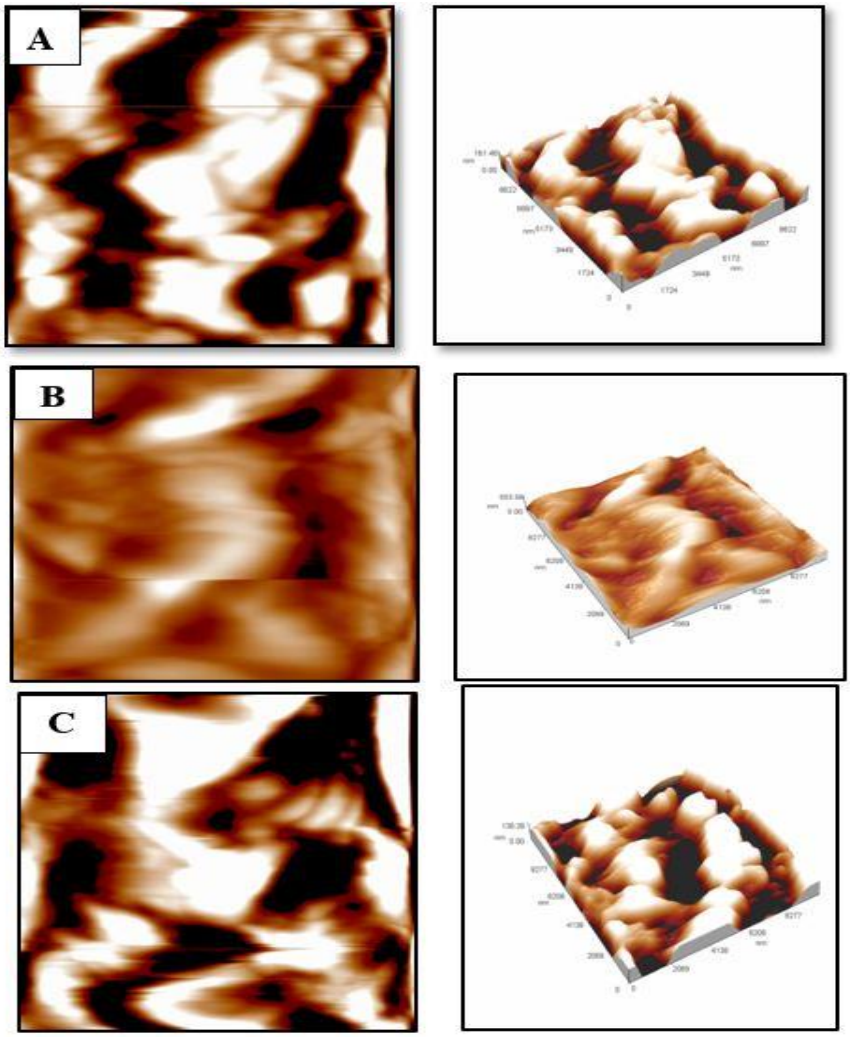
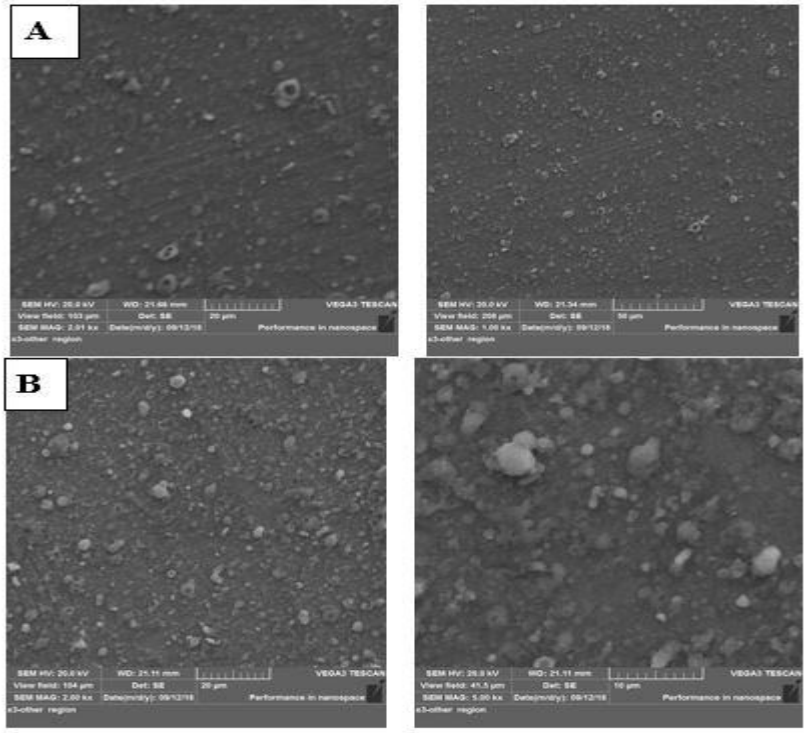


Figure (5): AFM Pattern of Samples at (A) B1, (B) B2 and (C) B3



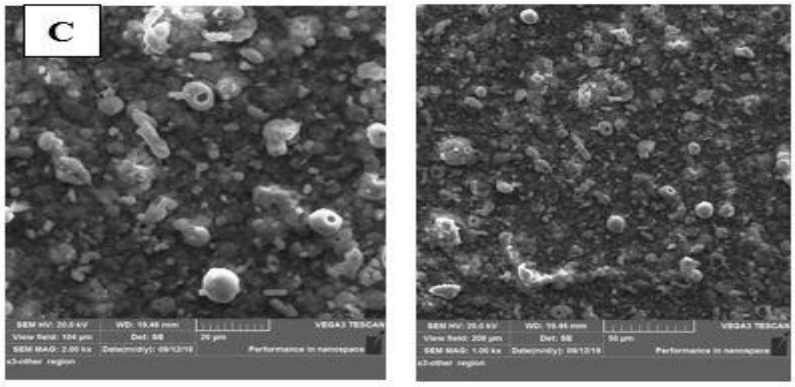


Figure (6): SEM Micrographs of Samples (A) B1, (B) B2, and (C) B3

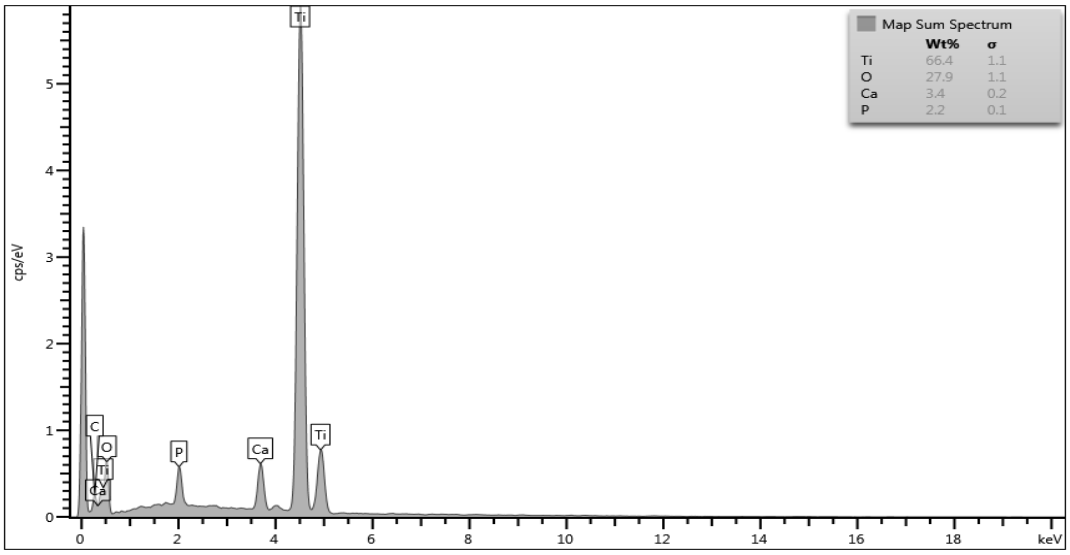


Figure (7): Results of EDX Analysis for B1 Sample

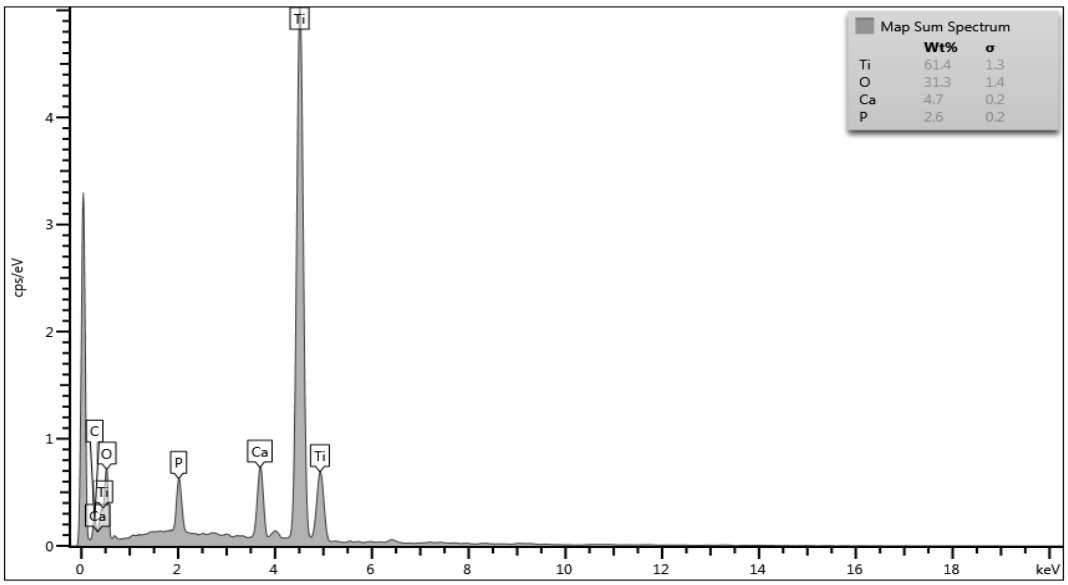


Figure (8): Results of EDX Analysis for B2 Sample

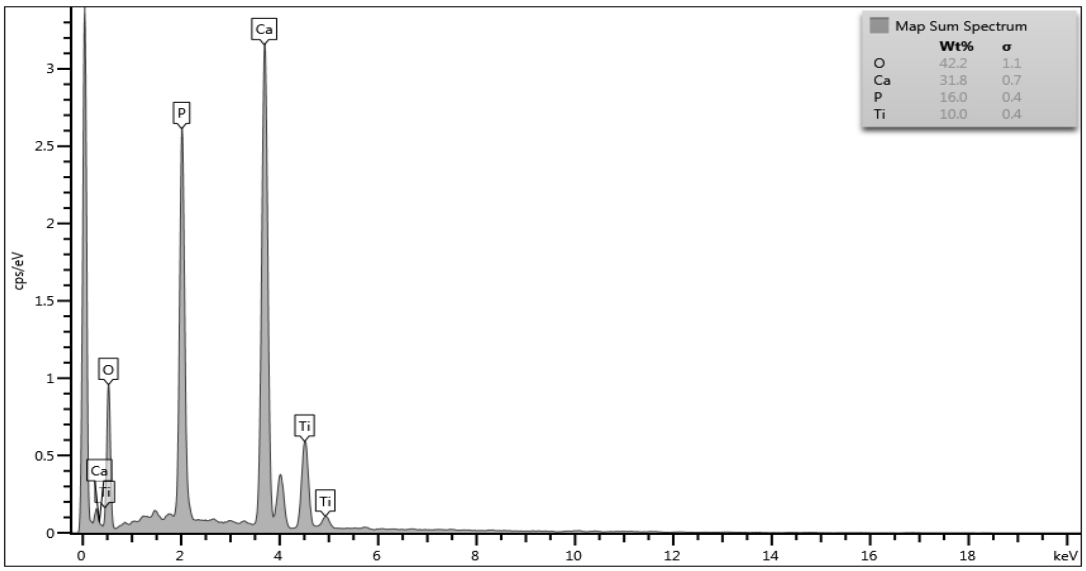


Figure (9): Results of EDX Analysis for B3 Sample

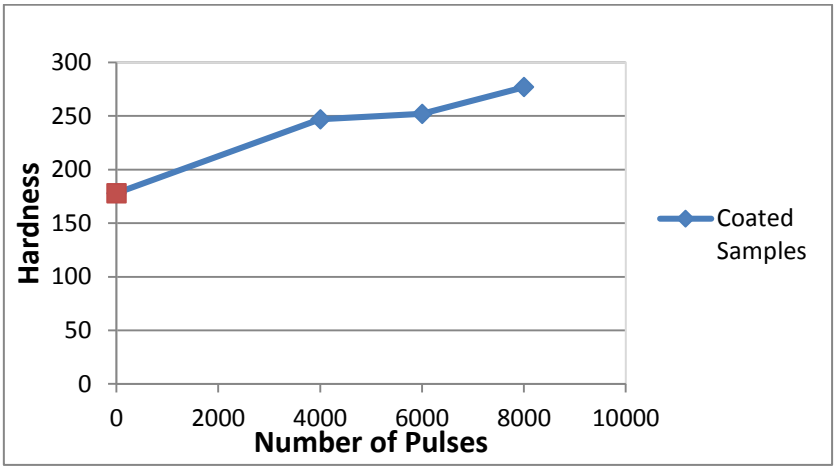


Figure (10): The Effect of Laser Pulses on HA coating Hardness (Hv).

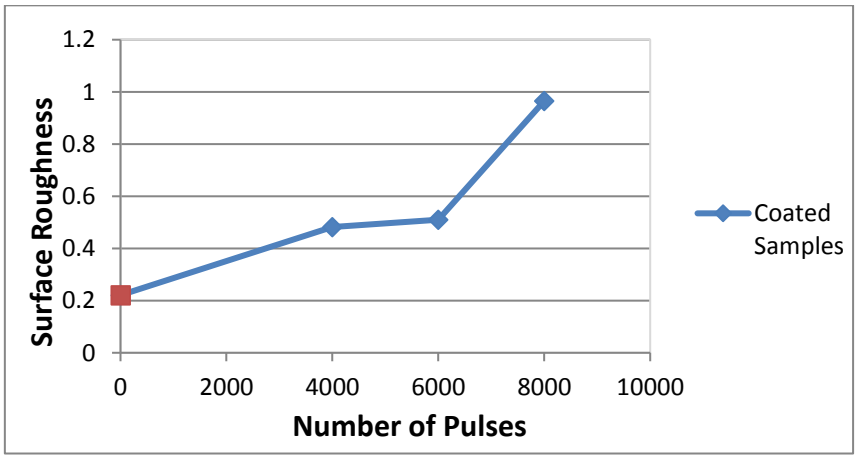


Figure (11): The Effect of Laser Pulses on Surface Roughness of HA coating.

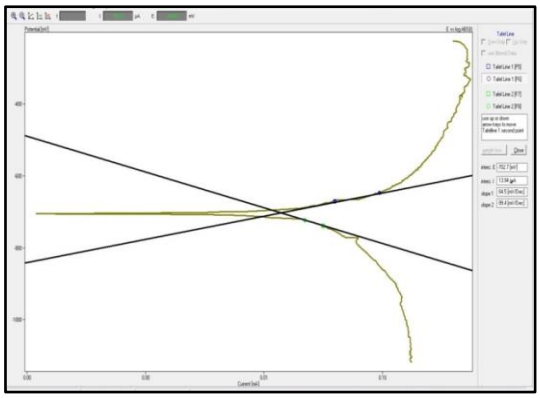


Figure (12): Potentiostatic Polarization for A Sample in Hank's Solution at 37 °C

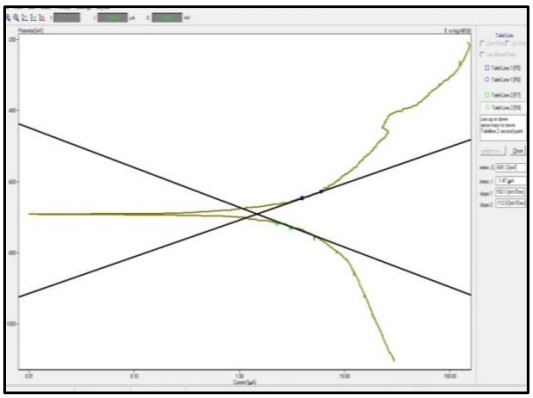


Figure (13): Potentiostatic Polarization for B1 Sample in Hank's Solution at 37 °C

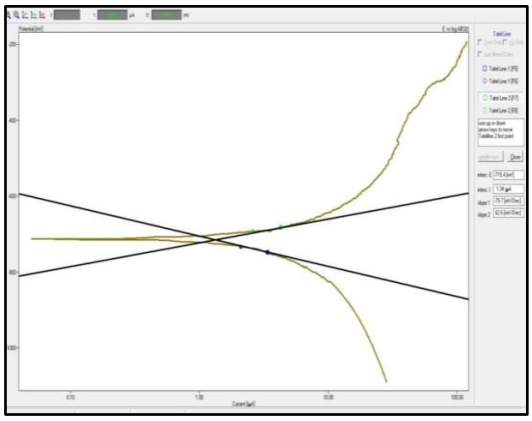


Figure (14): Potentiostatic Polarization for B2 Sample in Hank's Solution at 37 °C

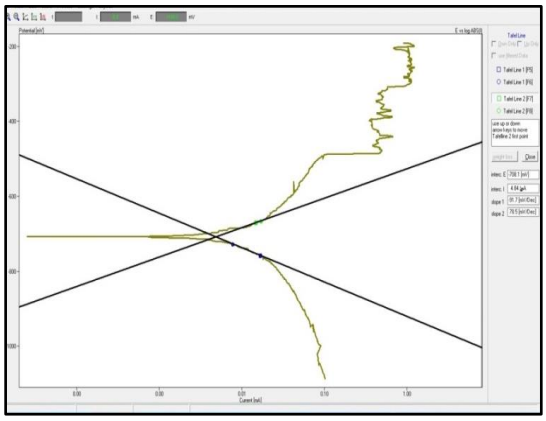


Figure (15): Potentiostatic Polarization for B3 Sample in Hank's Solution at 37 °C

CONFLICT OF INTERESTS.

- There are no conflicts of interest.

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تحري خصائص طبقة الهيدروكسي أبتايت المترسبة على عينات من التيتانيوم بتقنية الترسيب بالليزر النبضي

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الخلاصة

بحثت هذه الدراسة وأعدت طلاء مركب للمزروعات الجراحية باستخدام المواد السيراميكيه النشطه بايولوجيا (bioactive) مثل الهيدروكسيابتايت كطلاءات للمزروعات المعدنيه التي تشجع النمو الطبيعي للعظام عند نقاط التماس بينها وبين العظم الحي. ان الهيدروكسيابتايت ($Ca_{10}(PO_4)_6(OH)_2$) او فوسفات الكالسيوم يستخدم كطلاء للمعادن بسبب توافقيته الحياتيه الممتازه كما انه ذو هيكل مشابه للانسجه الصلبه في جسم الانسان. إن تطبيق الطلاء HA على ركائز التيتانيوم أنتجت باستخدام تقنية الترسيب بالليزر النبضي (Pulsed Laser Deposition). في هذا البحث استخدم (HA) كهدف (Target) تم كبسه عند ضغط (150MPa) مع حجم حبيبي مقداره (2.745 µm) واستخدامه في عملية الطلاء بواسطة تقنية(PLD). أجريت عدة اختبارات لتوصيف طبقة الطلاء مثل XRD وSEM وAFM وEDX لتحديد كمية كلا من الكالسيوم (Ca) والفسفور (P) في طبقة الطلاء. وبعدها تم اختبار الصلاده وخشونة السطح لطبقه الطلاء (HA). تم اجراء اختبار التآكل باستخدام طريقة استكمال منحني تافل في محلول Hank's solutionلكل النماذج المطليه وغير المطليه، حيث حصلنا في هذا الاختبار على تحسن كبير في مقاومة التآكل للعينات المطليه عند عدد نبضات 4000 نبضة بمقدار 99.88%.

الكلمات الدالة: طلاء، هيدروكسي ابتايت، مزروعات، التوافقية الحياتية، النسيج الحي، الترسيب بالليزر النبضي.