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RESEARCH ARTICLE

Diagnostic of Plasma Spectral and Development Ti6Al4V Alloy with Deposition Titanium Layer by R-F Sputtering

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Abstract

Ti6Al4V became the most favourable Titanium alloy due to its attractive mechanical and physicochemical properties and is currently extensively used e.g. in biomedical industry this work studies surface modification of Ti6Al4V to use in the implant bone. RF- sputtering is used for coating Titanium on the material Ti6Al4V alloy. The aim of this work is the power parameter (different power), used the ration power (50 watt, 100 watt, 150 watt and 200watt) the experimental Diagnostic of the coated layers by spectroscopy, FESEM and the average particle size (8.3-13.11) .The result show different colour layers, the TiN layer was shown the relationship between the electron temperature with the different power and electron density of TiN layer on Ti6 Al4V alloy with the different power (50,100,150and 200) Watt And during see the same figure the average particle size is equal for all picture between (8.3- 13.11) in the range of 100nm. The cumulative mass loss of the deposited coating specimen was about 3.71649mg for optimization samples TiN layer. The average hardness of the coating is about (264 to 331), which is higher than that of the substrate. The increases of average hardness depend on the different power.

Keywords: *Sputtering, Titanium, Homogenies, Implant, FESEM, Distribution, Surface modification and power.*

Introduction

Titanium and its alloys have been successfully used in the process of dental implants, joints and spine stabilization because of their good biological compatibility, corrosion resistance and mechanical properties. However, a large amount of debris caused by corrosion due to the poor performance of the Titanium alloys leads to toxic interaction with the physiological environment surrounding implantation and aseptic loosening of the implants and / or bone decomposition [1].

Other surgical alloys have shown better corrosion and adhesion properties than Titanium alloys. [2, 3] Thus, since its introduction in the early 1950s Ti6Al4V, Titanium alloys became the most favourable [4, 5]. The Ti-6Al-4V alloy has a unique advantage as it has the attractive properties and potential for work together (which allows it to be produced in all types of mill products of different sizes), its manufacturing capability well (allowing the mill products to be manufactured in complex equipment) And the experience of production and commercial availability leading to reliable and economic use. Thus, Ti-6Al-4V became the standard alloy wrought alloys which must be compared to other alloys when choosing Titanium alloys (or one custom design) for a particular application. Ti-6Al-4V is also a standard alloy selected for castings which must show superior strength. So that it has been evaluated in P / M treatment. Ti-6Al-4V will remain the most widely used Titanium alloy for many years to come [6, 9].

Material and Method

Titanium Sheet Target

Sputtering (stander) Titanium target with diameter (50 mm) and thickness (3 mm).

Ti6Al4V alloy as a Substrate (Product Description)

2pcs 8mm diameter 500mm length Grade 5 gr5 Titanium Ti stick-6AL-4V bar titanium alloy rod BT6 TAP6400 for medical treatment , **Item Specifics:**

- Torque capacity: gr5 Ti
- Model number: 8mm diameter
- Material: gr5
- Structure: gr5 Ti
- Coatings: gr5 Ti

Experimental Part

Ti-6Al-4V alloy samples were used in plasma sputtering (3 x 10 x 10) mm using a diamond cutter (Struers, Denmark). Ti-6Al-4V alloys were manufactured by grinding and polishing mirror respectively, using the Struers-RotoPol-21 system, Denmark. The SiC sheet was used in the flowing steps to grind samples: (120, 180, 240, 320, 500, 600, 800, 1000 and 1200µm of grain size).

Samples polished with DP-suspension for (15, 9, 6, 3.1µm) then a cloth port is used. The samples enhancing was done made by the same machine with Alumina powder (0.51µm) and red DP-Lubricant. Polished samples were cleaned with acetone-trichloroethyl acetone solution. The dilute samples were washed with deionized water, dried and stored in a dissociator above the silica gel pad and were used for plasma cures, microscopic development and electrochemical investigation. The cleaned specimens were stored on the sample holder and placed on the cathode plate to disperse the glowing plasma. The system was pumped down to the base pressure (about 10-4 mbar).Argon gas was introduced into the room (chamber). DC discharge of argon fluorescence (argon plasma) was discharged and put on to each sample for one hour. The sample has been cleaned with a break until all the small arcs have disappeared and a uniform glow can be seen throughout the sample. Plasma treatment of argon was used only as a standard measure to clean the surface of Ti alloy samples, reducing and removing the original oxide and the contaminated layer.

The argon gas (Ar) was introduced in the evacuated chamber and the flow rate was adjusted until the pressure was stabilized at the desired pressure, the sputtering specimens were cooled in the same gas. The cooling in the same environment is mostly performed to inhibit the oxidation of the samples. The plasma treatment of Ti-6Al-4V samples was done under a N2 (1-3mbar) pressure. The condition plasma Rf-sputtering includes in the below Table (1) and Figure (2) show the coating layer on the Titanium samples and the examination plasma diagnostic parameter (temperature, density) for TiN, argon by spectrometer device SV2100 (Kmac, Korea) and the partical size by Hitachi (S-4160) scanning electron microscope the magnification power continuous form 6x to 100,000x . Used Micro hardness measurements digital micro hardness tester (HVS-1000, Japan) and tests a pin-on-disc machine (Wear and Friction Monitor Tester TR201made by M/S DUCOM, Bangalore, INDIA) conforming to ASTM G 99 standard.

Table 1: The condition plasma Rf-sputtering with different power (50,100,150and 200) Watt

Material	Pressure 1 Torr	Pressure 2 Torr	Load (power) W	Density kg/cm^3	Temperature C°	Time coating Hours
Ti	8.2×10^{-5}	1.3×10^{-2}	50	4.5	Room temperature	1h
Ti	510×10^{-5}	2.47×10^{-2}	100	4.5	Room temperature	1h
Ti	3×10^{-5}	2.19×10^{-2}	150	4.5	Room temperature	1h
Ti	1×10^{-5}	1×10^{-2}	200	4.5	Room temperature	1h

Result and Discussion

Diagnostic Spectral Plasma

The diagnostic of material by spectral of plasma is very important to determine the experimental standard spectral of material used (Argon and titanium). The actually ration of material in the coating layer. Figure (1) shows the comparation between the spectral standards of material coating and the experimental material (ArI, ArII and Ti for different power) taken by RF plasma spectral, the different types of material (stander of argon gas, titanium pure and spectral R-F plasma) .In this figure the Titanium peak is proportional with the power, the more the power is large the peaks becomes.

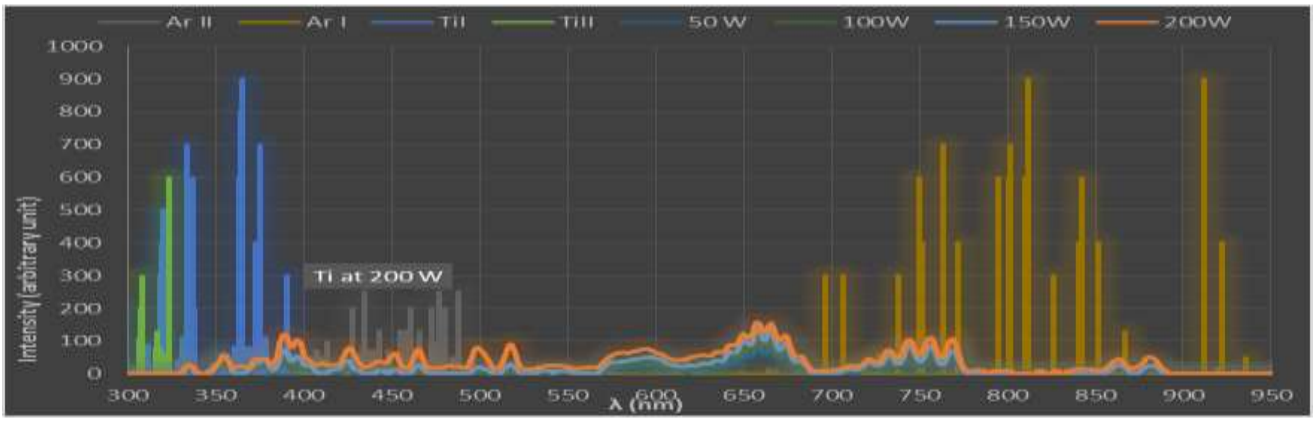

Figure. 1: Spectral plasma diagnostic with different power

Plasma Parameter (Temperature and Density Electron) by Boltzmann Method

Table (2and 3) shown the relationship between the electron temperature with the different power and electron density with the different power. It is observed from figure 2 and 3 the semi same behaviors of T_e and N_e which increased in the optimum power 200 watt. And this is noted in figure (2and 3) shown the relationship between the electron temperature with the different power and electron density of TiN layer on Ti6 Al4V alloy with the different power (50,100,150and 200) Watt. It is observed from figure 2 and 3 the semi same behaviors in the two curve of T_e and Ne which increased in the optimum power 200 watt.

Table 2: temperature with different power

No.	Describes samples	Power ratio (Watt)	Temperature (eV)
1	TiN coating	50	0.6612
2	TiN coating	100	0.6725
3	TiN coating	150	0.6835803
4	TiN coating	200	0.7702265

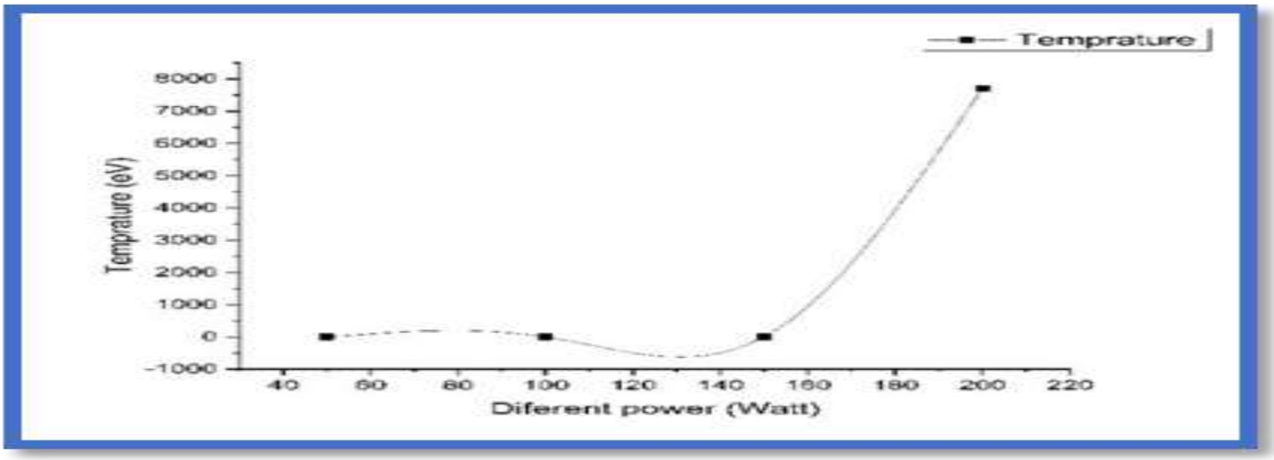

Figure.2: Electron temperature with different power (50,100,150 and 200)

Table 3: show the temperature with different power

No.	Describes samples	Power ratio (Watt)	Density
1	TiN coating	50	$1.2*10^{21}$
2	TiN coating	100	$1.21*10^{21}$
3	TiN coating	150	$1.26*10^{21}$
4	TiN coating	200	$1.33*10^{21}$

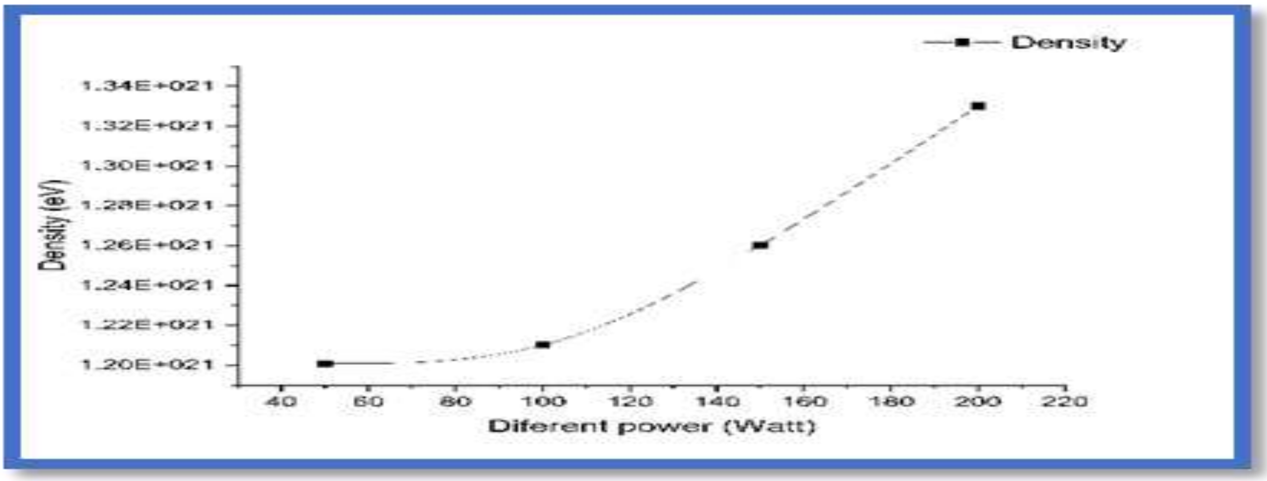

Figure.3: Electro density with different power (50,100,150 and 200)

Atomic Force Microscopic Analysis (AFM)

Atomic force microscopic analysis is ideal for quantitatively measuring the nano metric dimensional surface roughness and for visualizing the surface nano-texture of the deposited layers. Measuring the surface texture of TiN thin layers with horizontal length scale of 256 pixel and a vertical length scale of 250 pixel is critical for optoelectronic applications. Two-dimensional (2D) and three - dimensional (3D) AFM images of TiN layers at different power from 50 to 200 Watt is shown in Figure (4and 5). The surface roughness of TiN layers increased from 2.141 to 5.773 Pixel as the power increased from 50 to 200 Watt. The roughness parameters are estimated by the analyzing the topography scans of the sample's surface. The surface profile parameters includes average roughness (Ra), root mean square roughness (Rq), maximum peak to valley height (Rt), ten point average roughness (Rz), size dimension (SD) shows in Table (4) for different power (50, 100, 150 and 200) for TiN layer.

Figure 4: AFM (3D) images of Ti N layers at different power (50, 100, 150 and 200)

Figure 5: AFM (2D) images of Ti N layers at different power (50, 100, 150 and 200)

Table 4: the parameter of Atomic force microscopic analysis (AFM) at different power (50, 100,150and 200)

Power	R_a	Mean	R_t	R_q	R_z	SD
50	2.141	131.92	31.80	15.44	28.35	8.9929
100	4.006	111.23	68.79	28.89	58.70	13.661
150	4.585	136.77	65.99	33.06	56.81	12.5
200	5.773	115.58	86.06	41.63	75.56	18.334

Field Emission Scanning Electron Microscopy

The study of FESEM (Fig. 6, 7, 8, 9) of TiN films that grew at a flow rate of 1.25 has a very homogeneous surface and may be much stressed where contrast cannot be found, so the growth mechanism is not well clear. The analysis of the microscopic structure of Ti6Al4V by FESEM was performed by FESEM results. The graph shows the recorded microscope of the film grown between (50, 100, 150 and 200) watts of film with excellent texture, high homogeneity and dense microscopic structure with the polishing of grain produced under plasma bombardment. The improved density, uniformity.

The particles appeared flattened and interconnected. Also, the grains were uneven in size. At a capacity higher than -200 watts (Figure 8), there was a form of spherical formation that was dense, void and lop-sided throughout the existence of a few large randomly dispersed large pellets.

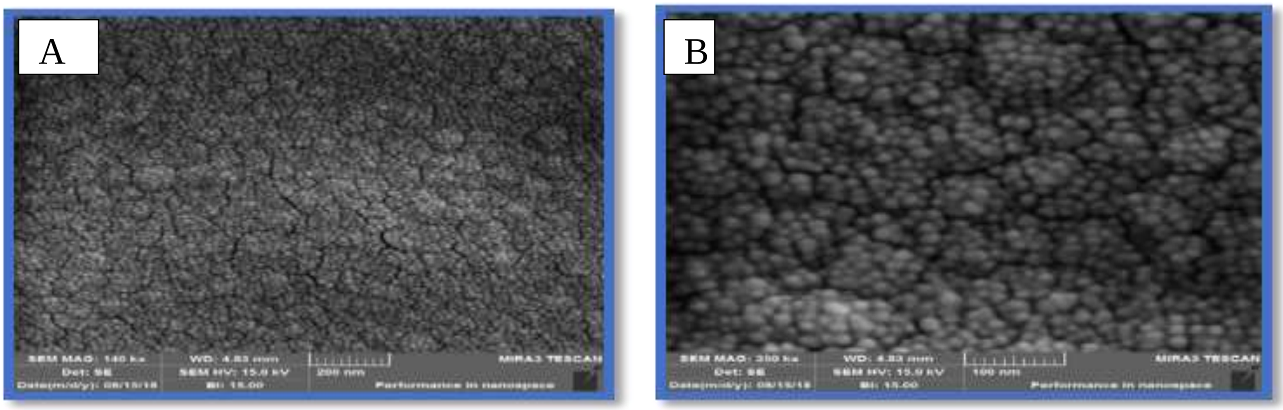

Figure.6: FESEM Images for TiN Layer by power 50W a. 200nmscale b.100nm scale.

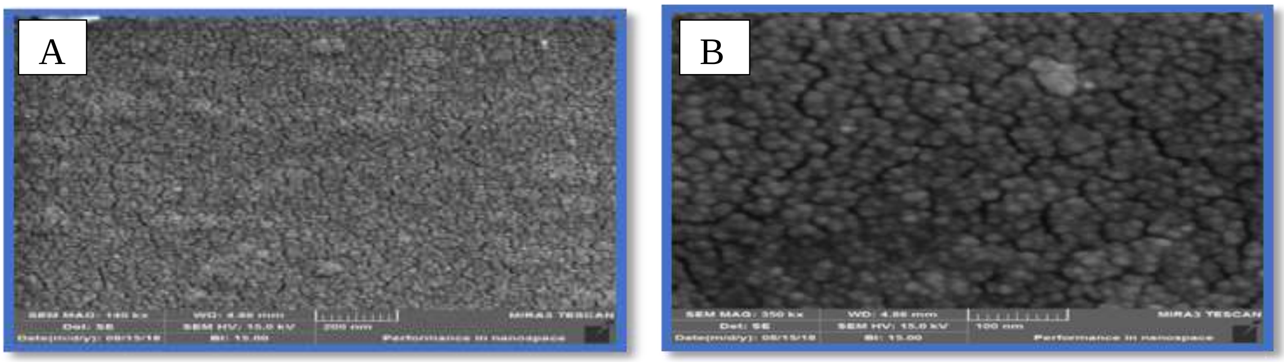

Figure.7: FESEM images for TiN Layer by power 100W a. 200nmscale b.100nm scale

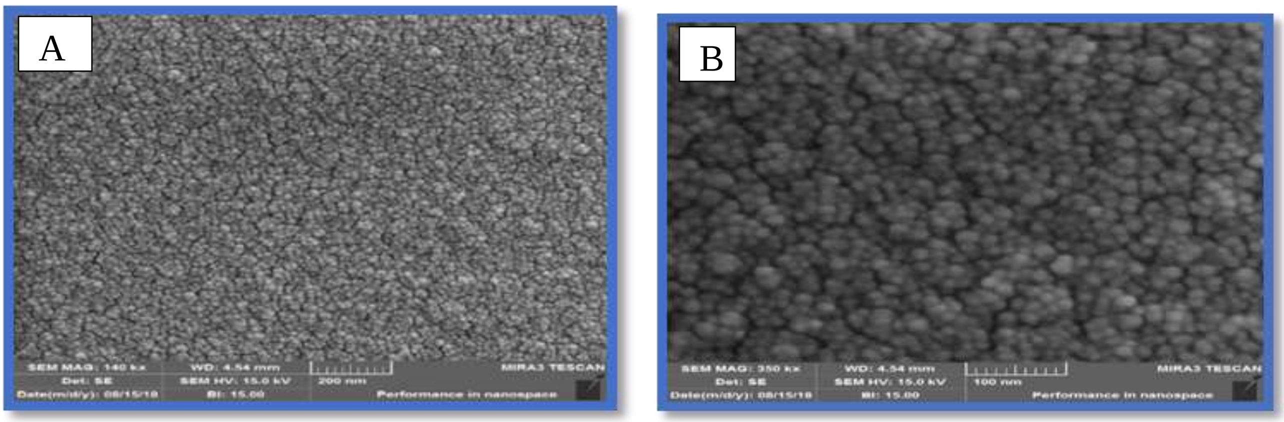

Figure.8: FESEM images for TiN Layer by power 150Watt a. 200nmscale b.100nm scale

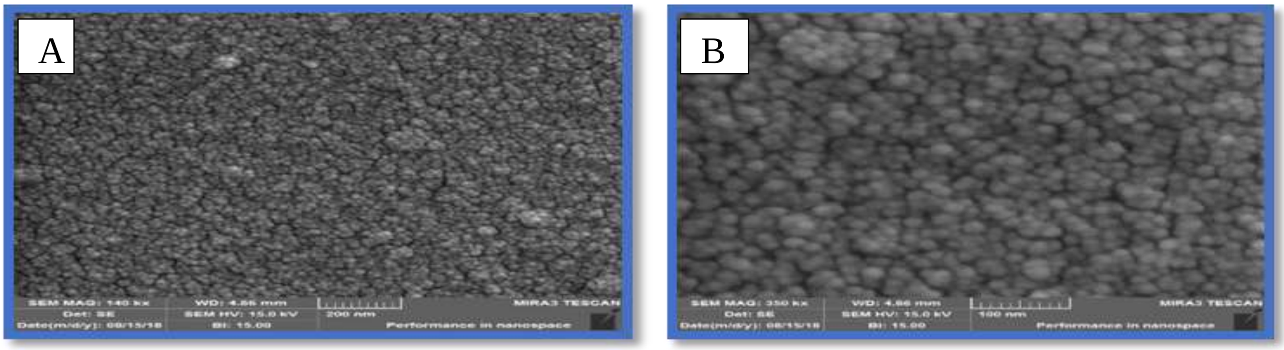

Figure.9: FESEM images for TiN Layer by power 200W a. 200nmscale b.100nm scale

Particle size by FESEM

In this test show the measurement of particle size coating on the Ti6Al4V the Figure (10, 11, 12 and13) given the particle size in Nano this test sure the material and method used is good.

And during see the same figure the average particle size is equal for all picture between (8.3- 13.11) in the range of 100nm.

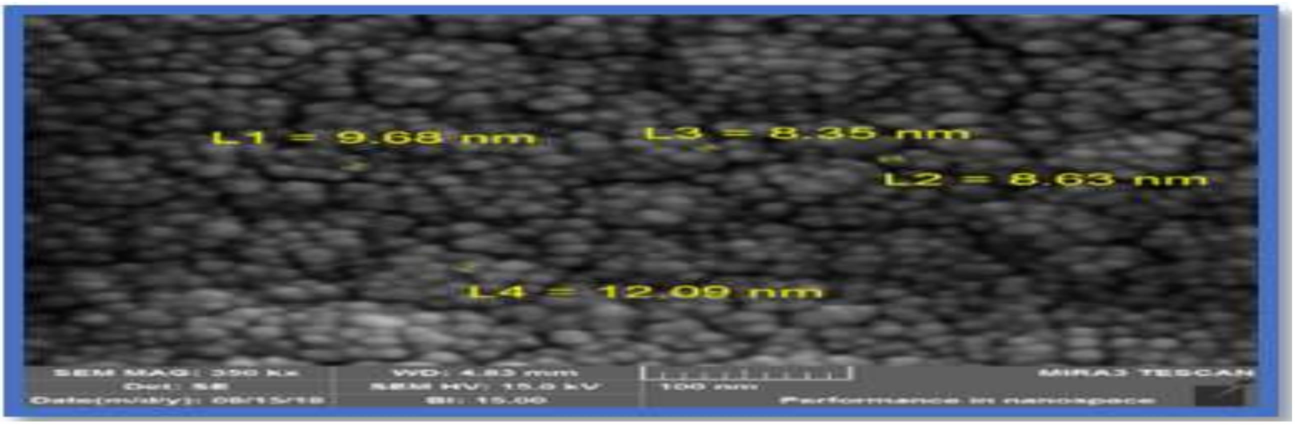

Figure.10: FESEM images for Ti N Layer by 50W

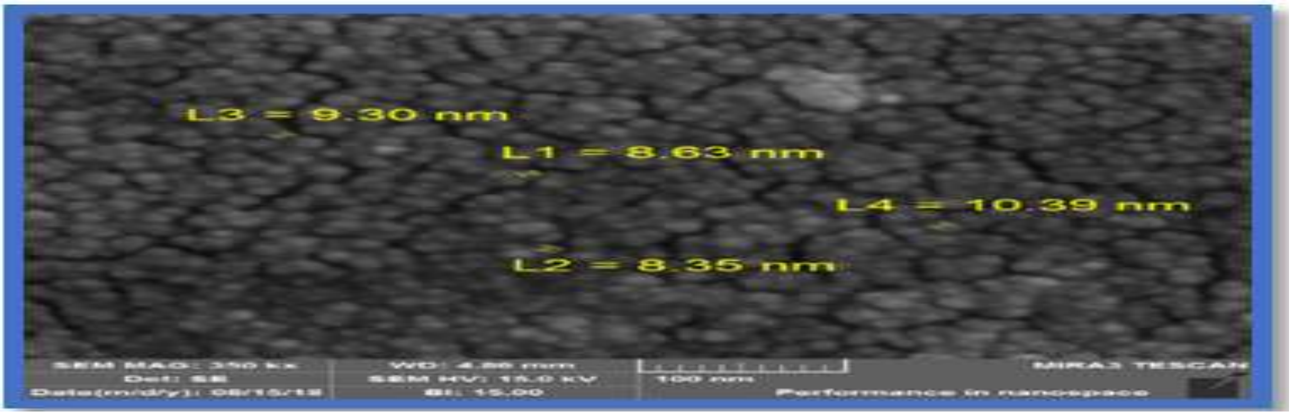

Figure.11: FESEM images for TiN Layer by 100W

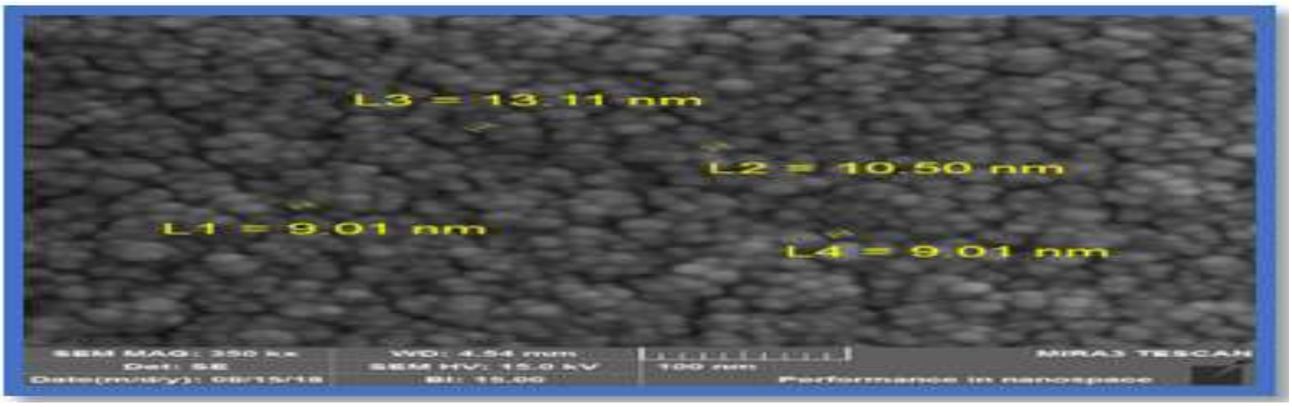

Figure.12: FESEM images for TiN Layer by 150W

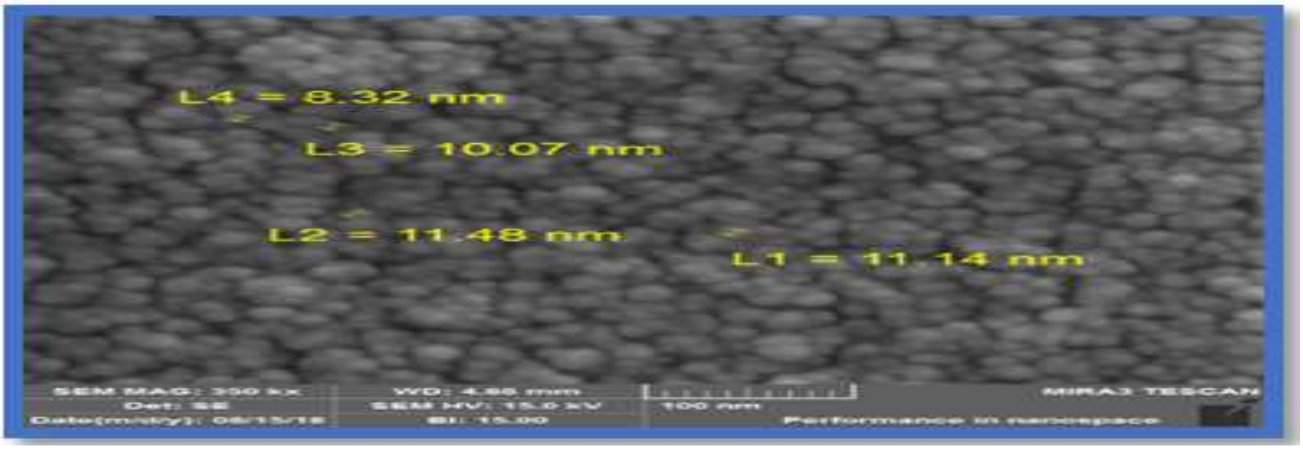

Figure.13: FESEM images for TiN Layer by200W

Mechanical Properties

Wear Test

The variations of the mass loss with test time (sliding distance) for the deposited coating and the substrate are shown in Figure (14, 15, 16, 17 and 18). The cumulative mass loss of the deposited coating specimen was about 3.71649mg for optimization samples TiN layer, while the mass loss of the substrate specimen was about 1.04069 mg. For convenience, the mass loss has been translated to the wear rate, which is also shown in Figure 12a with a special coordinate axis on right part. The wear rate of the deposited coating is about 2.66 Loss weight higher than that of the substrate, which implies that the deposited coatings possess better wear resistance than Ti6Al4V substrate.

Table 5: the result of weight wear test with different power (50,100, 150 and 200)

Sample Code(Power)	Ball Initial Weight, mg	Ball Final Weight	Ball Weight Loss, mg	Disc Initial Weight, mg	Disc Final Weight, mg	Disc Mass Loss, mg
M-1- stander	0.69200	0.69210	-0.10	1.04145	1.04069	0.76
M-2-50 Watt	0.65924	0.65900	0.24	4.05525	4.05469	0.56
M-3-100 Watt	0.72317	0.72134	1.83	3.21268	3.20996	2.72
M-4-150Watt	0.74903	0.74885	0.18	3.71759	3.71649	1.10
M-5-200 Watt	0.72664	0.72553	1.11	1.30213	1.30272	-0.59

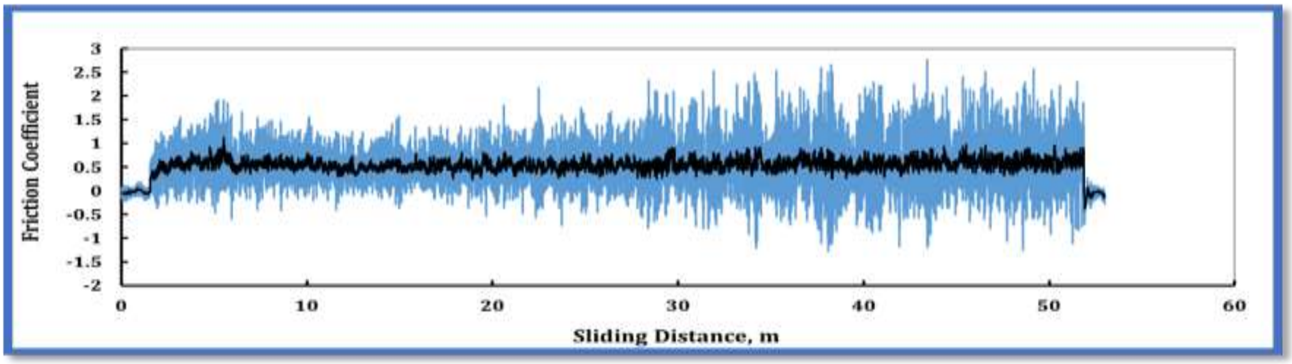

Figure 14: The wear test for deposited coating and substrate: fraction coefficient for stander

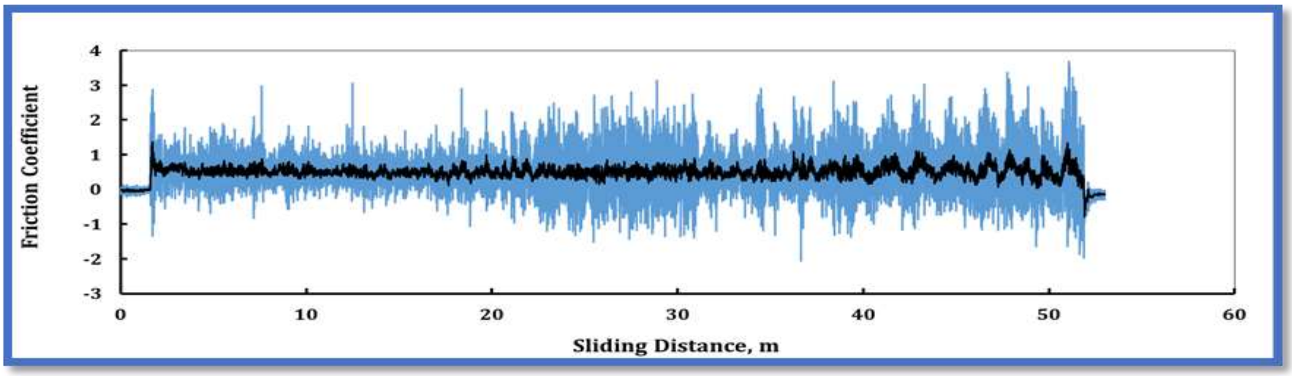

Figure 15: the wear test for deposited coating and substrate: fraction coefficient at 50 Watt

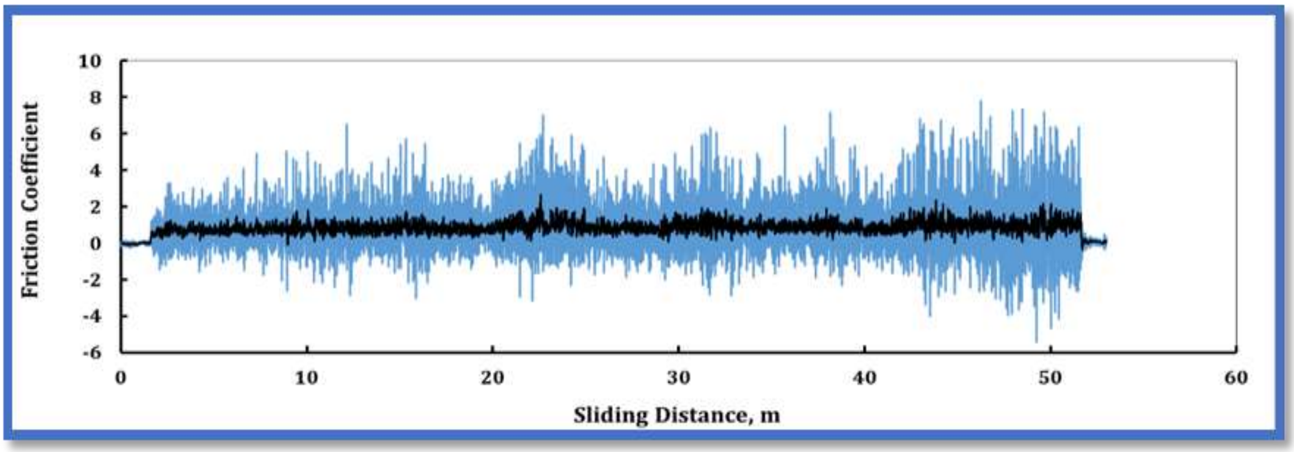

Figure 16: the wear test for deposited coating and substrate: fraction coefficient at 100 Watt

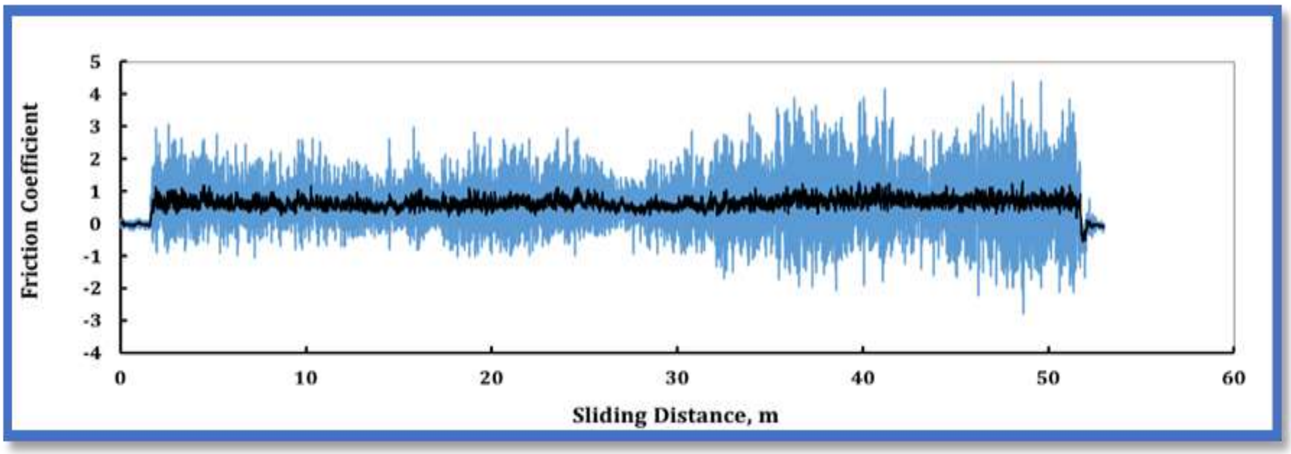

Figure 17: the wear test for deposited coating and substrate: fraction coefficient at 150 Watt

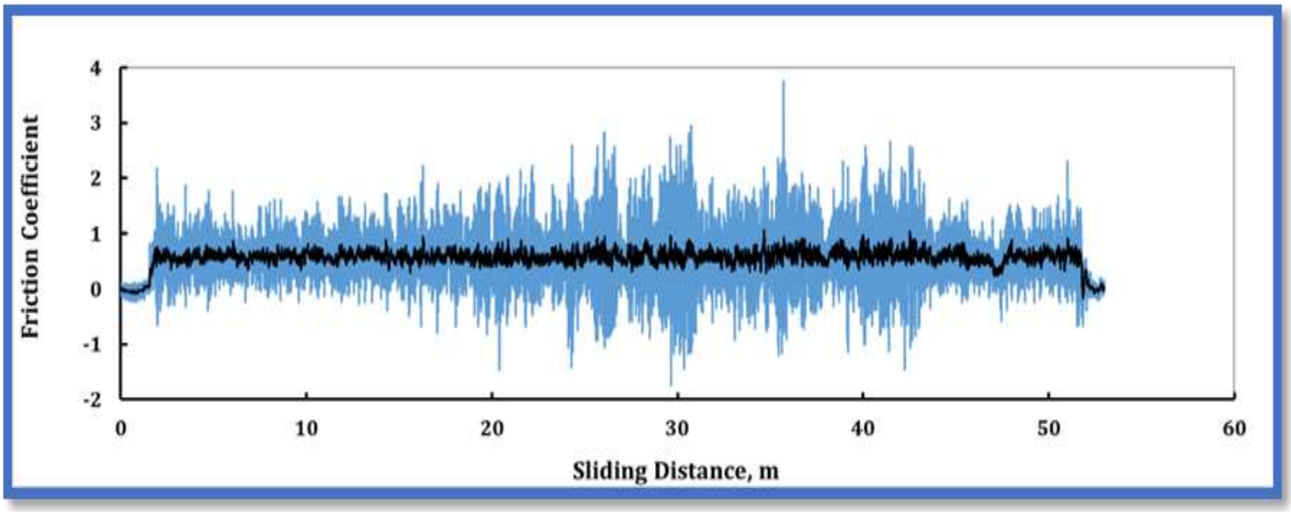

Figure 18: the wear test for deposited coating and substrate: fraction coefficient at200 Watt

Hardness Test

The micro hardness was tested on the samples Ti6Al4V alloy of the TiN layer. The distribution curve is shown in Figure (10). It can be seen that the hardness distribution of the deposited coating is even. The average hardness of the coating is about (264 to 331), which is higher than that of the substrate. The increases of average hardness depend on the different power shows in the Table (6).

Table 6: the result of hardness test with different power (50,100, 150 and 200)

Sample Code	First Hardness	Second Hardness	Average Hardness
M-1-stander	252.1	276.2	264.2
M-2-50 Watt	293.0	305.0	299.0
M-3-100 Watt	335.0	327.9	331.5
M-4-150 Watt	304.0	310.0	307.0
M-5-200Watt	281.0	295.0	288.0

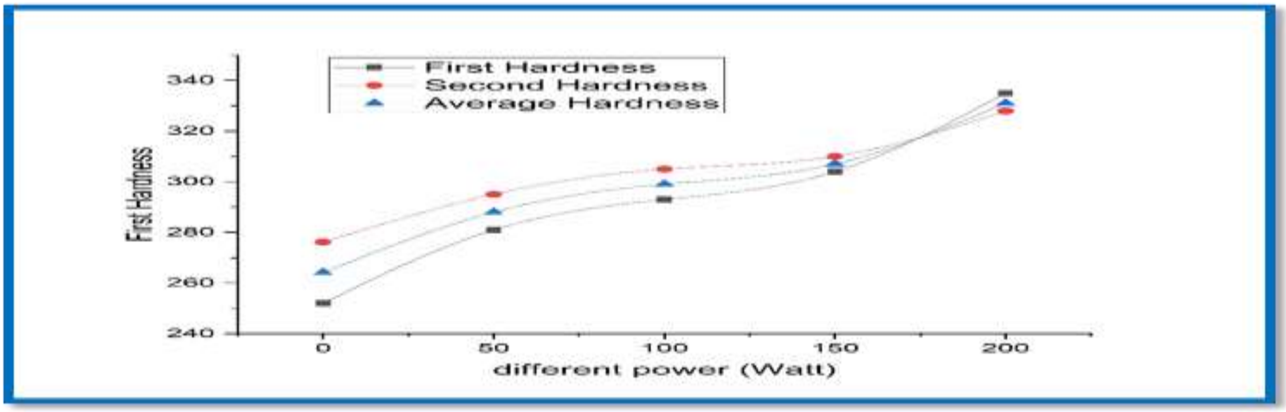

Figure 19: first, second and average hardness with different power (50,100,150 and 200) Watt

Conclusion

In this study used the optimization method (RF-sputtering) coating titanium on Ti6Al4V alloy for surface modification all result TiN coating when the high power the coating is good layer product show in the FESEM measurement the particle distribution on the surface is homogenous (good arrangement) also AFM and the optimization spectral plasma diagnostic for Ti6Al4V alloy at 200W is better than less power and appearance the mechanical properties (wear and hardness) same behaviour at 200 W.

References

1. HY Li, ZG Zha (2012) Research progress of the wear particles-induced aseptic prosthesis loosening, chin. Tissue Eng. Res., 16: 9059-9063.
2. JO Galante, J Lemons, M Spector, PD Wilson Jr, TM Wright (2015) The biologic effects of implant materials, J. Orthop. Res., 9: 760-775.
3. GA Afolaranmi, J Tettey, RMD Meek,* MH Grant (2008) Release of Chromium from Orthopedic Arthroplasties, J. Bone Jt. Surg. Br., 2: 10-18.
4. E Atar, ES Kayali, H Cimenoglu (2008) // Surf Coat Tech., 202: 45-83.
5. A Zhecheva, W Sha, S Malinov, A Long (2005) // Surf Coat Tech., 200: 21-92.
6. MJ Donachie (2000) Titanium: A Technical Guide, 2nd Edition.
7. LJ Martínez-Miranda, Y Hu (2001) Mol. Cryst. Liq. Cryst. Sci. Technol., Sect., A 364: 419.
8. M Abdullah1, 2• Farah T Mohammed Noori2• Amin H Al-Khursan1 (2016) Opt. Quant. Electron., 48:15.
9. Farah TM Noori (2016) Experimental, Kinetics, Isotherms and Quantum Chemical Calculations; Corrosion Inhibitor for Mild Steel in Acidic Medium: Wulfenia journal Klagenfurt austia, 23: 2.
10. SB Al-Baghdadi, FTM Noori, WK Ahmed, AA Al-Amiery (2016) Journal of Advanced Electrochemistry, 2(1): 67-69.