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Research Paper

Corrosion of Platinum Nanocoatings Thin Films Deposited by Sputtering Deposition Method for Medical Applications

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Abstract

Platinum (Pt.) thin films have been deposited by Magnetron Sputtering Deposition method with 25 nm and 50 nm thickness by used power RF for deposited 5 nm Titanium (Ti) firstly then used DC for deposited Platinum (Pt) on Ni-Cr alloy substrate. SEM/EDX and Ellipsometry Spectroscopy have been used to investigate the morphology, element analysis, nanothickness and roughness of the thin films respectively. Electrochemical corrosion tests have been used to analyze the localized corrosion resistance by used cyclic polarization, Tafel extrapolation and open circuit potential (o.c.p) methods on artificial saliva solution at 37 ±1 ºC. The results show the ability to deposit 5 nm of Ti before deposited of Pt. film to improve the adhesive of the films, nanostructure morphology of the Pt. film without defect or microcracks, nanocoatings thickness of Pt. film, and high localized corrosion resistance in artificial saliva corrosive media.

Keywords: *Atomic absorption spectroscopy, Corrosion Resistance, Platinum, Sputtering Deposition, Artificial Saliva, Titanium, Thin Films*

1. Introduction

The natural and synthetic materials are contacted with living tissue of biological systems called biomaterial [Kamachi U 2003, Sujata K 2012], It's used for more than one hundred years, where at 1895 the academic Lane presented the first metal plate for bone fracture fixation [Miao Yu 2014, Hendra Hermawan 2014]. Biomaterials science is physical, chemical and biological knowledge of any materials interacted with the human body, furthermore to the biocorrosion resistance which is the first main point should be approved for any type of biomaterial using surgically in a human body. Also, saliva, food, bacterial microflora, temperature, and mechanical forces are the main factors in the oral cavity specifically [Joanna Mystkowska 2018]. Titanium and its alloy, Cobalt Chromium alloys, Stainless Steel 316L, Nickel-Chromium alloys are most biomaterials common used surgically [Nitech R.2012, Kotoka Ruben 2013, Davis J. R 2000]. Nanocoating is a term used when the coating thicknesses or the structures is between (1-100) nm (nanometer or nanostructure scales). Nanocoating thin films are a synthesis of coatings with different thickness to improve properties that are typically not found in conventional coating methods by used nanocoating facilities such as atomic layer deposition and sputtering deposition and pulsed laser deposition [Kiyotaka Wasa 2004, Injeti G 2008, Aliofkhazraei Mahmood 2011, Sami Abualnoun 2015]. Magnetron sputtering system is one of several different technical methods used to deposit thin films with good fabrication and characterization [David Joseph 2010, John D 2005]. In this work, we focus on depositing of 25 nm and 50 nm thin films of Pt. on the Ni-Cr-Mo alloys for producing uniform films with homogeneity, also for investigating the morphology and demonstrating the localized biocorrosion resistance of Pt. films electrochemically which are using in medical applications.

2. Materials and Methods

In this study, medical grade samples of Ni-Cr alloy with 10 x 10 mm^2 dimensions were used, as shown in Table (1). The surfaces were cleaned and prepared by acetone firstly than by ethanol and deionized water (AED) as a first preparation method.

Table 1: Chemical composition of Ni-Cr alloy used

Elements	Mo	Cr	Fe	Cu	Ni	
Weight	9.94	20.9	2.4	0.13	Bal.	

Pure Ti - target and pure Pt- target (99.995%, ISO 900: 2008) from Kurt J. lesker company with power RF for Ti and DC for Pt. were used in Magnetron Sputtering. The vacuum base pressure was 2.8 $x10^{-7}$ Torr, rate 2 nm/min for Ti and 6 nm/min for Pt. Also, the argon gas used at 20 sscm, the pressure during deposition of 4m Torr to deposited ~5nm Ti as an adhesion layer before depositing the Pt. The System is calibrated with different targets and power. So the adjustment graph is used to estimate the power and time needed for depositing a given film's thickness. Scanning electron microscope/ energy dispersion spectroscopy (SEM/EDS) in the University of Missouri-Columbia-USA with an Acc. Voltage: 5.0 kV Take Off Angle: 36.9 deg. & live time of 30 sec to investigate the defect of films deposited. Conductive carbon adhesive is used to mount the samples on the holders. For controlling the process of imaging Smart SEM-software are used. AFM in a laboratory of Nanotechnology center - the University of Technology is used for investigating the surface roughness of samples and glass. Spectroscopic Reflectometer system kind TFP-robe with software presented for measuring of Pt. film thickness of glass deposited instantaneously with the samples of Ni-Cr alloy substrate. WINKING M Lab 200 Potentiostat from Bank Elektronik in Materials Eng. Dept. / the University of Technology is used for analyzing and study the electrochemical corrosion resistance, where the coated and uncoated samples are working electrode, the

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Platinum metals are the auxiliary electrode, and as a reference electrode used saturated calomel electrode (SCE). Electrochemical software completed in electrochemically at a scan rate of 0.5mA.sec^{-1} . Table (2) shows the composition of artificial saliva used as a corrosive media.

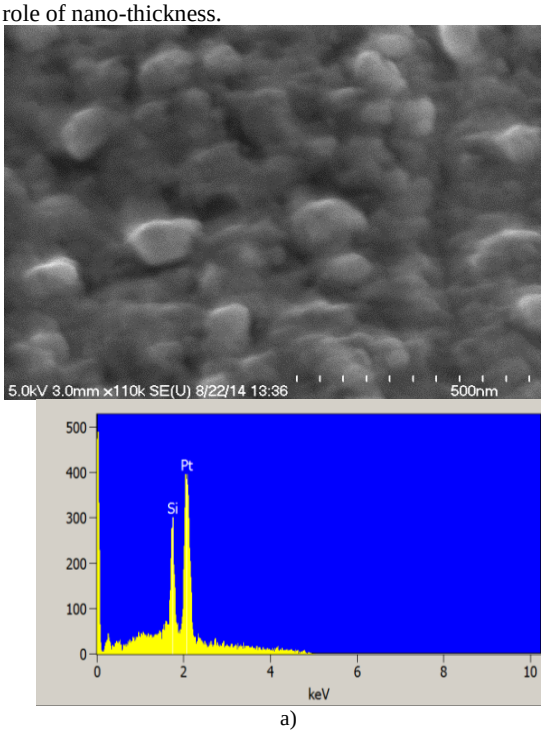
Table 2: Artificial saliva Composition [Haitham M. 2016, Muna Khethier 2017]

Element	Conc.(g/L)
$CaCl_2.2H_2O$	0.906
KCl	0.4
$Na_2S.9H_2O$	0.005
NaCl	0.4
$NaH_2PO_4.2H_2O$	0.69
Urea	1

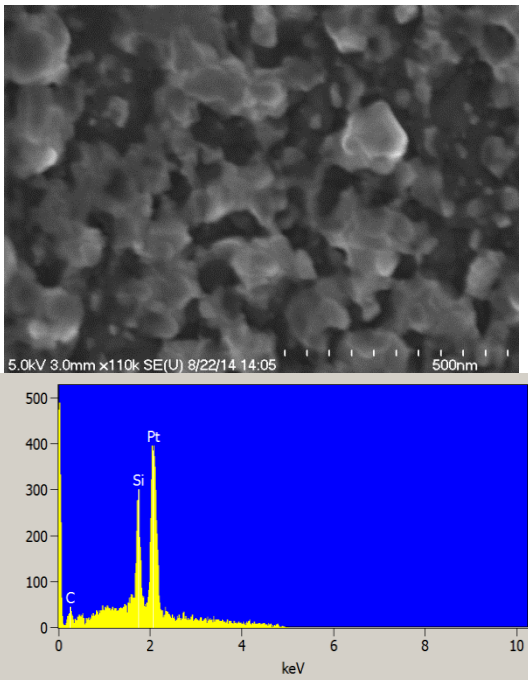
Cyclic polarization, Tafel extrapolation and open circuit potential (E_{ocp}) in artificial saliva solution at 37 ±1 ºC were used in this study. Corrosion current density (i_{corr}), Corrosion potentials (E_{corr}), breakdown potential ($E_{break.}$), and re-passivation potential ($E_{Rep.}$) are results obtained. Atomic absorption spectroscopy (AAS) was used to analyze the results of corrosion elements after tests at Ibn-Sina state company/ Ministry of Industry and minerals / Baghdad.

3. Section headings

Fig. (1 a & b) shows SEM micrograph of the 25 nm and 25 nm of Pt. thin films deposited on Ni-Cr-Mo alloy substrate. It can be observed that the thin films are free of a crack and there isn't any defect. Where the surface morphology seems clear and deposited films have a nanoparticle size as shown at 500 nm magnifications. The EDS Figures show peaks of Pt. element which is thin film deposited, and Si element which is Si-wafer used as a substrate. At EDS of 25 nm, as shown in Fig. (1 a), the carbon peak appears furthermore to the Pt. and Si peaks. This is related to the conductive carbon used as adhesive to mount the samples on the holders but this peak didn't appear for 50 nm thickness which related to the role of nano-thickness.

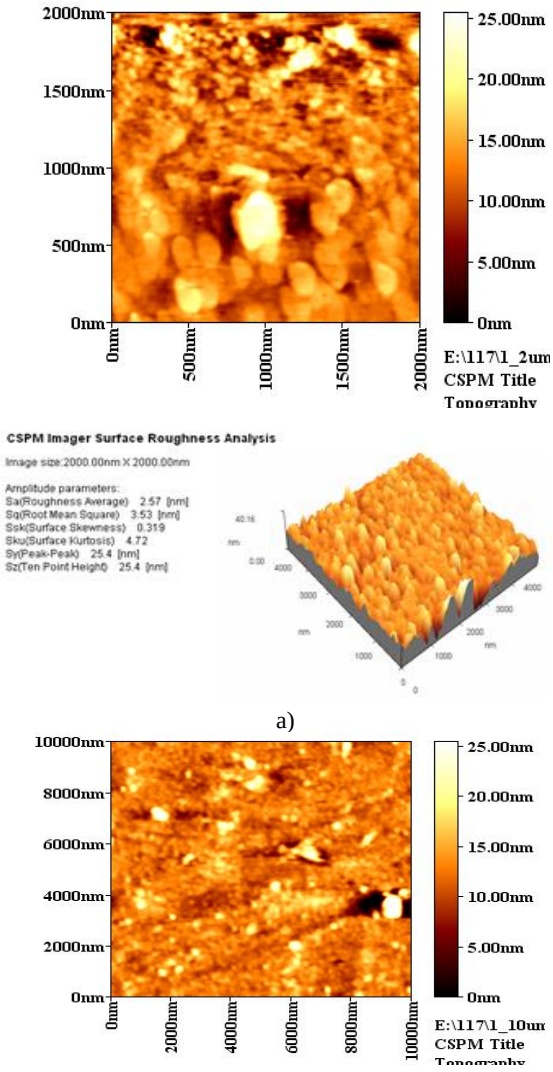


a)

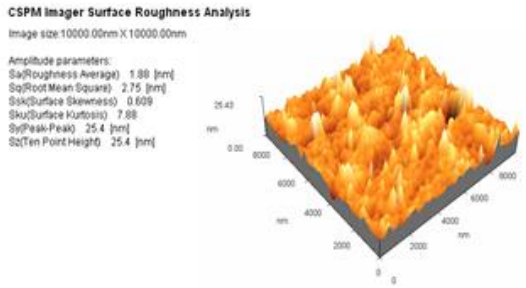


b)

Fig. 1: SEM/EDS micro-graphs of 25 nm (a) and 50 nm (b) of Pt. thin films.



a)

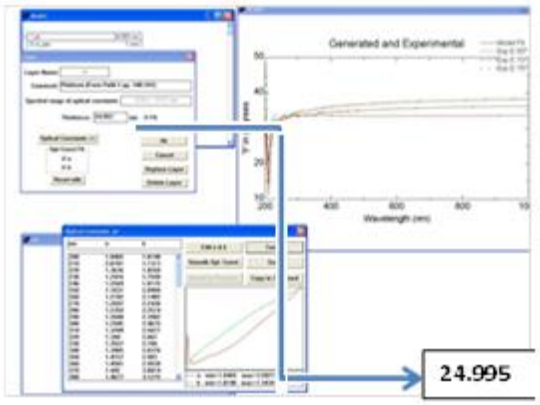


b)

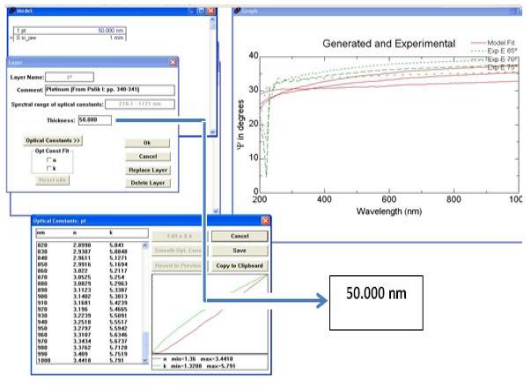
Fig.2: The results of AFM images for two and three dimension of Pt. nanocoating thin films with; a) 25 nm, and b) 50 nm.

Fig. (2) Shows AFM images of the 25 nm and 50 nm of Pt. thin films. It appears that the morphology of Pt. film was significantly different with growing the thickness, where the roughness reductions from 2.57 nm to the 1.88 nm as shown in Fig. (2). The increase of Pt. thickness leads to increase in the homogenous and smoothness of the thin films surface as an indication to a role of nano-thickness effect when increased from 25 nm to the 50 nm.

Fig. (3) shows the ellipsometry spectroscopy results of samples nanocoated with 25 nm and 50 nm of Pt. thin films. It seems that the thickness was 24.993 and the refractive index (n) was 1.4677, but for samples nanocoated with 50 nm the thickness was 50.00 nm and the (n) was 3.4418. It is determined from the results of ellipsometry spectroscopy tests which obtained that Pt. thin films are characterized by uniformity with excellent control over the thickness.



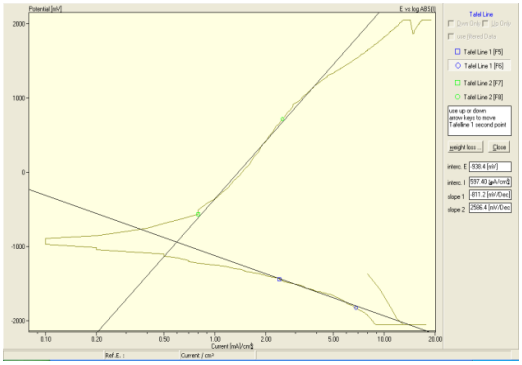
a)



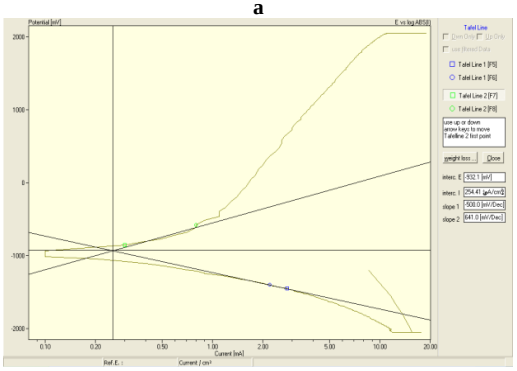
b)

Fig.3: Ellipsometery spectroscopy results of Pt. nanocoating thin films with;a) 25 nm , and b) 50 nm

Table (4) and Figures (4 and 5) shown the corrosion results for uncoated and nanocoated specimens by Pt. thin films with 50 nm in artificial saliva corrosive media at 37 oC . The Eo.c.p became more stability after nanocoated where was (–675mV) for uncoated sample and decreased to (-180 mV) after nanocoated thin films of Pt. Corrosion current densities (i corr.) were 597.4 µA.cm-2 for uncoated sample and 254.41 µA. cm-2 for nanocoated thin films of Pt.



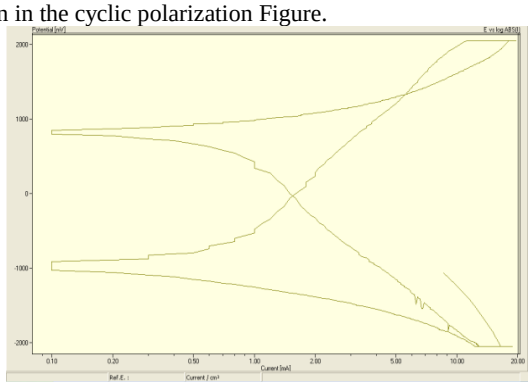
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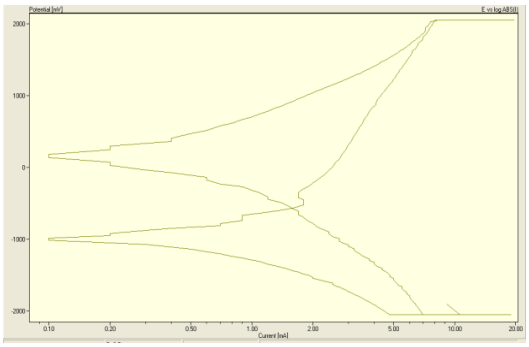
b

Fig. 4: Tafel results of Ni-Cr alloy substrates; uncoated sample, b) nanocoated with 50 nm of Pt.

The more biocorrosion resistance for nanocoated thin films substrate is due to the stability of Pt. films in the reactions of corrosion electrochemically, which leads to protecting the surfaces in the corrosive media from the corrosion reactions. The E corr. of a nanocoated substrate is more than that of uncoated; it establishes to the low tendency of Pt. thin films to the corrosion reaction. The active-passive metals for localizing corrosion are depended on E break. and E rep. Although the E break. of nanocoated and uncoated is equal to (+2000 mV) , but there isn't E rep. value for nanocoated with Pt. thin films compared with that of uncoated alloy, this means no corrosion pits and more localized corrosion resistance. Also, the deceases the hysteresis loop of nanocoated samples with 50 nm of Pt. compared with uncoated alloy as shown in the cyclic polarization Figure.



a



b

Fig. 5: Cyclic polarization results of Ni-Cr alloy substrates; a) uncoated sample, b) nanocoated with 50 nm of Pt.

Table (4): Results of electrochemical corrosion tests.		
Parameters of Corrosion	Uncoated Ni-Cr-Mo alloy	Nanocoated with 50 nm of Pt.
+b (mV/Dec.)	+2586.4	+641
-a (mV/Dec.)	-811.2	-500
E break. (mV)	+2000	+2000
E corr (mV)	-938.4	-932.1
E o.c.p (mV)	- 675	-180
E rep.(mV)	+1350	No
i corr (µA.cm-2)	597.4	254.41

The result of AAS for corrosion solution after corrosion test are arranged in table (5) for uncoated and nanocoated with 50 nm of Pt. Where the results show elements of Ni, Cr, and Mo in different concentrations for corrosion solutions of uncoated sample compared with no elements results for corrosion solutions of nanocoated samples with 50 nm of Pt. thin films.

Table 5: AAS result of corrosion solution after corrosion test.

Materials element	Account (ppm)	
	Uncoated samples	Nanocoated with 50 nm of Pt.
Cr	0.003	No results
Mo	0.195	
Ni	0.007	

4. Conclusion

From deposited thin films of Pt. by using sputtering deposition method on Ni-Cr alloys specimens concluded the excellent homogeneity of Pt. thin films, good thin films thickness control, and high thin films area uniformity have been obtained. Furthermore to different nano-particles shape and size obtained depending on a thickness of thin films deposited (25 & 50 nm). Also, Pt. thin films of 50 nm have a great effect on improving the electrochemical corrosion resistance of coated Ni-Cr alloys compared with the uncoated samples in artificial saliva solution at 37±1 ºC without any metallic ions of Ni, Cr, and Mo are noted in the artificial saliva used as a corrosive media.

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