Biocompatibility, Bioactivity and Corrosion Resistance of Stainless Steel 316L Nanocoated with TiO₂ and Al₂O₃ by Atomic Layer Deposition Method

Muna Khettier Abbass ¹, Sami Abualnoun Ajeel ² and Haitham Mohammed Wadullah³

¹ Dept. of Production Engineering and Metallurgy, University of Technology, Baghdad, Iraq, Email: dr.munakabbass@gmail.com
² Dept. of Production Engineering and Metallurgy, University of Technology, Baghdad, Iraq, Email: samiabualnon2@yahoo.com
³ Engineering Technical College, Northern Technical University, Mosul, Iraq, Email: dr.haitham@ntu.edu.iq,

*Email: dr.munakabbass@gmail.com

Abstract. Atomic Layer Deposition (ALD) method has been used to synthesis nanocoatings thin films of Alumina, Titania, and Alumina/Titania multilayer on stainless steel AISI 316L at 250 °C deposition temperatures for the medical applications. SEM and EDX have been used to characterize the morphology of the films and the element analysis of the alloys and the thin films respectively. Open circuit potential, potentiostatic polarization (Tafel extrapolation) and cyclic polarization methods have been used to study the corrosion resistance of the films in Simulation Body Fluid (SBF) at 37 ± 1 °C. Immersion test in SBF for 2 weeks at 37 ± 1 °C has been used to determine the biocompatibility of the films. Numbers of colonies and diffusion zone methods have been used after cultured non-pathogenic E-coli bacteria to demonstrate the bioactivity (toxicity effects) of the thin films. The SEM morphology observations show different particles shape and size of the Alumina (nanotubes shape around 10-20 nm in size) and Titania films (cauliflower particles shape around 20-50nm in size). The results also show that the corrosion resistance can be effectively enhanced by thin films, multilayer proved to be more corrosion protection than single layers, and Alumina has better corrosion resistance than Titania.

Keywords: Alumina, Titania, stainless steel 316L, ALD, thin films, corrosion resistance
1. Introduction

Corrosion is a complex multi-factorial phenomenon that depends on metallurgical, geometric, mechanical, and chemical parameters of the solution environment. Corrosion is the first main consideration for any type of metals to be used in surgical implants. Biomaterial is any natural or synthetic material that interfaces with living tissue and/or biological systems [1, 2]. Biomaterial science is physical and biological study of materials and their interaction in the body. Historically, biomaterials have been used for more than 100 years when the researcher Lane introduced the first metal plate for bone fracture fixation in 1895 [3, 4]. Austenitic Stainless Steel (316 and 316L), Cobalt-Chromium-Molybdenum alloy and Titanium and its alloys are commonly used in metals implants [5, 6, 7]. In 1920s, 18-8 stainless steels had been introduced and immediately attracted the interest of the clinicians due to the grouping of good mechanical properties and far-superior resistance to corrosion, in that time, and were used in a wide range of biomedical applications [8]. The corrosion resistance of stainless steel 316L is due to Chromium and Molybdenum contents, where chromium allows the formation of chromium oxide ($Cr_2O_3$) intentionally on the surface of the metal when chromium in the surface layer reacts with oxygen, adherent and coherent oxide thin film (passive layer) envelops the surface and serves as a barrier to corrosion reactions [9, 10]. But, chromium oxide passive film formed on the surface of the implant metal is unstable and prone to localized corrosion in longtime implant applications due to harsh biological effect because the human body is a very complex and aggressive electrolytic environment for biomaterials applications. Also, austenitic stainless steels are not susceptible to intergranular corrosion by the precipitation of chromium carbides at the grain boundaries ($Cr_23C_6$), and depleting of the chromium from it, sensitization [11, 12]. Xiao-Yan Zhang et.al [13] has been found that the improvement of 316L SS for orthodontic bow corrosion resistance was depended on the laser power from studied the effect of laser surface remelting on the corrosion resistance of 316L Orthodontic bows and improving its corrosion resistance from artificial saliva.

Nanocoating term is used if the coating thicknesses in nanometer scales (1-100nm), thin films, or the structures are in nanostructure scales. Thin films and substrate are synthesis system with different enhanced properties because thin films materials have vast capacities that do not typically found in conventional coatings [14,15]. Mohammed et al. [16] in 2018 used the electrophoretic deposition (EPD) to produce the nanohydroxyapatite layers on 316L stainless steel substrate. The EPD coatings were prepared by the deposition of hydroxyapatite (HA)-chitosan nanocomposites on different substrate roughness (polish surface, 220 grit SiC grind, and sand blast surfaces). The results showed the deposited coatings on sand blasted substrate has less porosity compared with the polish surface and 220 emery paper SiC grinding substrate respectively. They obtained a good bonding between substrate and coatings, since this bonding have an important role in biomedical applications especially in bone replacement applications.

Currently, nanotechnology enables the production of ultra-thin films and nanocoatings consisting of just one monolayer or a few atomic layers. Atomic Layer Deposition (ALD) Method is a layer-by-layer deposition technique, gas phase chemical process, and a unique method for depositing high quality thin films even with extremely complex shapes. Its special modification of the more common used chemical vapor deposition (CVD) technique [17].

The aim of the present work is synthesis of high quality nanocoatings thin films of Alumina, Titania, and Alumina/Titania multilayer on stainless steel 316L specimens by use ALD method, then characterization and evaluation the structure of the nanocoatings thin films, and study the corrosion behavior, biocompatibility and bioactivity of the thin films deposited.
2. Experimental Work

Stainless steel type 316L provided by Good-fellow corporation-USA with specimen’s area (20×20 mm²) and thickness 0.9 mm. Table 1 shows the chemical composition of AISI 316L. The samples were ultrasonically cleaned by acetone for 60 minute, washed by methanol and de-ionized water (AMD) to remove all of the organic contaminants before dried by hot air and load into the deposition chamber. Savannah Atomic Layer Deposition (ALD) reactor manufactured by Cambridge NanoTec Inc was used in Nanotechnology Core Facility (NCF) - University of Missouri-Columbia, USA to deposit Alumina (Al₂O₃), Titania (TiO₂) and Alumina /Titania multilayer.

| Table 1 | Standard and analytical composition of stainless steel AISI 316L |
|---------|---------------------------------------------------------------|
| Element (wt%) | Cr | Ni | Mo | C | Si | Mn | P | Fe |
| Standard | 16-18 | 10-14 | 2-3 | 0.03 | 0.75 | 2.0 | 0.045 | Bal. |
| Analytical | 16.5 | 10 | 2.0 | 0.027 | 0.375 | 1.32 | 0.045 | Bal. |

Titania was deposited by dimethylamido (TDMAT) precursors, chemical compound of Ti (NMe₂)₄. Alumina was deposited by Trimethylaluminum (TMA) precursors, chemical compound of Al (CH₃)₃. Nitrogen (N₂) was used as a carrier and purging gas. TDMAT, TMA precursors and Nitrogen gas were provided from Sigma–Aldrich (99.9%). Alumina/ Titania multilayer were by deposition of Alumina layer in the range of 25 nm using TMA and then above it deposited Titania layer in the range of 25 nm using TDMAT. H₂O precursors for each one was used, in the same vacuum chamber with continuous process and on different substrates. All the depositions were performed at a temperature of 250°C. The high temperature processes were chosen to obtain defect free, homogenous, and uniform layers in addition to a crystalline structure. The number of deposition cycles of each layer was calculated using a growth rate per cycle for each type which is ~0.39 Å/cycle for Titania and 1.07 Å/cycle for Alumina, as shown in table 2.
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Table 2  Parameters of deposition thin films.

| The parameters                     | Alumina | Titania |
|-----------------------------------|---------|---------|
| System-growth temperature.        | 250°C   | 250°C   |
| Heater 8 and 9 (deposition temp.) | 250°C   | 250°C   |
| Heater 6, 7 and 10                | 150°C   | 150°C   |
| N₂ gas flow                       | 20 sccm | 20 sccm |
| Pulse TMA (valve 3)               | 0.015 s, then wait 5s | 0.1 pulse , then wait 5s. |
| Pulse H₂O (Valve 0)               | 0.015 pulse , then wait 5s | 0.015 pulse , then wait 5s. |
| Growth per cycle                  | ~1.07 Å/cycle (Å/C) | ~0.39 Å/cycle (Å/C) |
| No of cycle                       | 480 cycle for 50 nm | 1284 cycle for 50 nm |

Ellipsometry Spectroscopic type WVASE32-USA instrument with software available for optical data analyses to measure the thickness and the refractive index coefficient (n) for thin films deposited on silicon wafer(100) in Nanotechnology Core Facility - University of Missouri-Columbia, USA. Incident and reflected angles between 65° to 75° were used at wavelengths from 2000 to 10000 nm. Cauchy model was fitted to guess the thickness by Cauchy software optimization.

SEM used is Hitachi S4700-Japan type with an operating voltage of 3.5-10.00 kV at resolution of 400 nm. Quanta 600F is a facility for Energy dispersive X-ray spectroscopy (EDX) with SEM machine used in order to find the element analysis of the thin films by using a working distance of 12 mm, accelerating voltage 10.0 kV and takes off angle 37.3 degree. SEM used in Electron Core Facility (ECF) - College of Veterinary Medicine in the University of Missouri-Columbia, USA.

For Electrochemical corrosion study, Potentiodynamic polarization type WINKING M Lab 200 Potentiostat/Galvanostat from Bank-Elektronik was carried out in Materials Engineering Dept.-University of Technology-Baghdad, Iraq. Simulation body fluid solution (SBF) used, as shown in table 3, at pH adjusted to 7.4 and temperature of 37±1°C. Tafel potential range was ± 250 V of the Ecorr for uncoated and coated samples. Electrochemical software used at 5 mV/sec scan rate and SCE in potentiostat. The main parameters that can be got from the potentiodynamic polarization curves are the corrosion potential (Ecorr) and the corrosion current density (icorr). The breakdown potential (Eo) and repassivation potential (Erep) were carried out by cyclic polarization at scan rate of 5 mV/s.

Corrosion rate is calculated depended on the current density the equation [18, 19]:

$$\text{Corrosion Rate (mpy)} = 0.13 \times i_{\text{corr}} \times \left(\frac{e.w}{\rho}\right)$$  \hspace{1cm} (1)

where; i corr ; is corrosion current density (µA.cm⁻²), e.w ; is equivalent weight (gm), and ρ ; is the density (gm/cm³). Mpy meaning mils per year (0.001 in ) as a penetration range.

Biocompatibility test was in Nanotechnology Core Facility - University of Missouri-Columbia, USA. It was done according to the ASTM F2475 and ASTM F981-04. The samples were immersed in a bio-cell, which was composed of container, pH-meter, thermocouple, hot plate magnetic stirrer, and Ringer solution at 37ºC. Duration of immersion was 2-week. In the end of immersion time, the samples were
raised, dried and investigated by SEM-EDX in Production Engineering and Metallurgy Department- University of Technology- Baghdad, Iraq.

Table 3 Chemical composition of simulation body fluid solution (SBF).

| Formula                  | gm. / liter |
|--------------------------|-------------|
| Sodium chloride (NaCl)   | 9.00        |
| Potassium chloride (KCl) | 0.43        |
| Calcium chloride 6H₂O (CaCl₂) | 0.24    |
| Sodium bicarbonate (NaHCO₃) | 0.20    |

Bioactivity test was investigated in Nanotechnology Center-University of Technology- Baghdad, Iraq. It was done according to ASTM F895 – 11 (standard test method for agar diffusion cell culture screening for cytotoxicity). One of the most important reasons for culturing bacteria in vitro is to study the toxicity effects of the films on the bacteria, the non-toxic film that is killed fewer cells of non-pathogenic bacteria, bioactive films. In this section, the toxicity effects of alumina, titania and alumina/titania multilayers thin films is evaluated by cultured the E-coli bacterial on the thin films at 37 °C for 24 hr.

For a Preparation of Bacterial Microorganism, Non-pathogenic Escherichia coli (E-coli) bacteria used in this test were selected from the collection of the laboratory of Biotechnology branch in Department of Applied Science/University of Technology. The bacteria were sub cultured on nutrient agar (N. agar), England Company, and incubated for 24 h at 37 °C. The Bacteria were suspended in 50 ml of normal saline to yield a bacterial suspension of around 10⁷ (CFU/ml), Number of Colonies, using plate count. Normal saline solutions used was 0.9% of NaCl, prepared in conical flask by adding 0.9g of NaCl dissolved in 90 ml of distilled water. The pH of the solution was 7 and sterilized by autoclave before used. Number of Colonies [(CFU)/ml] = (Number of colonies for each dilution) / (dilution factor)/ sample volume. Also, estimated the percentage reductions of bacteria using [20]:

\[ R = \left( \frac{(Z-A)}{Z} \right) \times 100\% \]  

Where Z & A is the number of live cell of bacteria per milliliter corresponding to the control samples and corresponding to the thin films samples cultured respectively. Diffusion zone method depended on the measured the area around the samples which has different color almost off white, number of bacteria died.

3. Results and Discussion

Table 4 shows the ellipsometry spectroscopy results of 50 nm of Alumina, Titania, and Alumina/Titania. For Alumina film, the number of cycles is 470 cycle, measured thickness is 47 nm, and refractive index is 1.638. The standard deviation in measured thickness compared to the theoretical thickness is about 1.5% for 50 nm of Alumina. For obtaining 50 nm of Titania, the number of cycles is 1284 cycle, measured thickness is 43.04 nm, and refractive index is 2.145. The standard deviation in measured thickness compared to the theoretical thickness is about 3.5% for the 50 nm of Titania respectively. Measured thickness is 49.6 nm for Alumina /Titania multilayer, standard deviation is about 0.2 % from the theoretical thickness, and the refractive index is 1.98. On the other hand the measured thickness for single layer is 26.99 nm for Alumina at 25 nm and 26.45 nm for Titania at 25 nm, a total of 53.33 nm. The presence of multiple layers introduces added complexity in the software modeling used to interpret the ellipsometer results because each layer has different refractive index as denoted in a previous study [21].
Table 4  Data results by using ellipsometer device for thin film deposited by ALD.

| Type               | 50 nm Alumina | 50 nm Titania | 50 nm Alumina /Titania |
|--------------------|---------------|---------------|------------------------|
| No of cycles (X)   | 480 (GPC=~1.07Å) | 1284 (GPC=~0397Å) | 250 cycle of Alumina+ 700 cycle of Titania |
| Theoretical Thickness | 50 nm       | 50 nm         | 50 nm                   |
| Measured Thickness | 47.3nm       | 43.04 nm      | 49.6 nm                |
| Standard deviation | 1.5%         | 3.5%          | 0.2%                   |
| Refractive index (n)| 1.638        | 2.145         | 1.98                   |

It is concluded from the results of ellipsometry tests that the obtained thin films are characterized by excellent control over the thickness and uniformity. The standard deviation of the theoretical thickness did not exceed 3.5 % as a larger deviation in measured thickness for Titania thin films with 50 nm thicknesses, and the smaller deviation is 0.2 for Alumina/ Titania. Furthermore, the refractive index for Titania is higher around 2.145, and the lower refractive index is 1.618 for Alumina. The refractive index obtained in the acceptable limit as reported by previous authors [22- 25].

Figure (1a, c, & e) shows the SEM results of Alumina, Titania and Alumina /Titania multilayer thin films deposited on stainless steel316L specimens. It can be observed from the SEM images presented that there isn’t any defect, micro crack and blistering phenomenon on the thin films for all thin films deposited. The thin films also have excellent homogeneity and large area uniformity. The uniformity of the thin films is related to the type of deposition method used, ALD, at high deposition temperature (250 ºC). The Alumina films have nanotubes particles shape with very fine spherical particles size between them around 10-20 nm, while the Titania thin films are similar to cauliflower particles in shape with different particles size ranging between 20-50nm. This means that the particles shape and size of the Alumina films are different from that of Titania films. These results are similar to that reported in the literature [24]. The particles of the Alumina /Titania thin films have the same shape and size as that of the Titania thin film, because it’s the upper layer.

From Figure 1 (b, d&, e) shows the EDX results, it can be observed that there are aluminum with oxygen peaks for 50 nm of Alumina, Titanium with Oxygen peaks for 50 nm of Titania, and Aluminum with Titanium with oxygen peaks for Alumina/Titania multilayer thin films.
Figure 1  SEM/EDX results of the thin film deposited on the stainless steel 316L:

a) SEM image 50 nm alumina  
b) EDX of 50 nm alumina  
c) SEM image of 50 nm titania  
d) EDX of 50 nm titania  
e) SEM image of alumina/titania multilayer  
f) EDX of alumina/titania multilayer
Figure 2 and table 5 show the OCP-time results for 316L stainless steel specimens, the potential is generally changed from initial positive value of 170 mV to the more positive value of 182 mV and then remains stable at this value for uncoated specimens. The improved corrosion behavior of stainless steel 316L can be attributed to Chromium which forms passive Chromium oxide film (Cr₂O₃) as confirmed by [17]. The potentials of the OCP curves also for coated 316L stainless steel specimens by thin films of Alumina, Titania, and Alumina/Titania multilayer generally shift toward more noble direction and then are stable continuously with time. From the results are concluded that the stainless steel 316L specimens have passive behavior and after deposit thin films the corrosion resistance improved.

![Figure 2](image)

**Figure 2** Open circuit potential results in SBF solution at 37 ºC ±1 for uncoated and coated Stainless steel 316L by alumina, titania and alumina /titania multilayer.

**Table 5** OCP potentials of uncoated and coated stainless steel AISI 316L alloy

| Type               | Uncoated Co-Cr alloy | 50nm Titania | 50nm Alumina | Alumina / Titania |
|--------------------|----------------------|--------------|--------------|------------------|
| Potential start (mV) | +182                 | +231         | +270         | +275             |

Figure 3 and table 6 show uncoated and coated stainless steel 316L by Alumina, Titania, Alumina /Titania for investigating the cathodic and anodic polarization curves in SBF solution at 37±1 ºC. The corrosion potentials results of uncoated stainless steel 316L specimens have less positive corrosion potentials values and larger current densities than specimens coated 50 nm of Alumina, Titania and Alumina/ Titania multilayer.
respectively. The rapidly converting of Cr\(^{6+}\) to Cr\(^{3+}\) of alloys containing chromium decreases the concentration of chromium ions at the surface and then increases the dissolution of metal atoms [22]. It means that the corrosion resistances of coated specimens are better than that for uncoated specimens and the protective coatings by thin films, 50 nm deposited by ALD at 250\(^\circ\)C, have effect on the improving the corrosion resistance. Multilayer of Alumina / Titania thin films deposited on stainless steel 316L specimens have a greater decreases in the corrosion current densities and increases in potential values. It means that the Alumina / Titania multilayer provided more protection in corrosion resistance than that of single layer of Alumina and Titania separate at 50 nm thicknesses, and Alumina film was more resistance than Titania. This is due to the chemically abrupt of the Alumina/Titania multilayer introduces as confirmed by reference [25]. The diffusion bonding between Alumina/Titania multilayers created chemically complex barriers being effective in the corrosion resistance [21].

| Condition                  | \(E_{\text{corr}}\) Mv | \(i_{\text{corr}}\) nA.cm\(^{-2}\) | -bc mV/Dec | +ba mV/Dec | C.R (mpy) |
|----------------------------|------------------------|-------------------------------------|-------------|-----------|-----------|
| Uncoated Ss 316L -alloy    | 180.9                  | 4.56\times10\(^{-4}\)              | 110.3       | 85.8      | 1.913     |
| 50 nm of Titania           | 200                    | 4.8\times10\(^{-1}\)               | 119         | 134       | 0.2       |
| 50 nm of Alumina           | 200                    | 4.8\times10\(^{-1}\)               | 188         | 179       | 0.17      |
| 50 nm of Alumina / Titania | 246                    | 3.9\times10\(^{-1}\)               | 88.6        | 178.6     | 0.16      |

Figure 3  Polarization results of coated Stainless steel 316L specimens with 50 nm of alumina, titania and alumina/titania in SBF solution at 37\(\pm\)1 \(^\circ\)C.
Figure 4 and table 7 show cyclic polarization curves for stainless steel 316L specimens without coatings and coated with 50 nm of Alumina, Titania and Alumina/Titania. It shows cathodic and anodic regions and returns to the cathodic region again. It also shows that at the anodic region has rapid increase in potential from critical potential until reaching breaking potential. Then the potential decreases until it intersects the curve at repassivation potential. The cyclic polarization curves results are agrees with the results reported by [26] for stainless steel 316L in simulating human body fluids. Smaller hysteresis loops are found in the results of 50 nm thin films which mean fewer tendencies to the localized corrosion and more resistance to pitting corrosion than that of uncoated samples. No hysteresis loops, higher breakdown and repassivation potential of Multilayer than that of single layer (Alumina or Titania) also indicated from the result which means more resistance to pitting corrosion. Corrosion rate calculated by current density is 1.913 mpy for uncoated sample and decreases after coated to 0.203 mpy for Titania film, 0.174 mpy for Alumina film, and 0.164mpy for Multilayer.

**Figure 4** Cyclic polarization results in SBF solution at 37±1 °C for:

a) Uncoated stainless steel 316L specimens;

b) Coated with alumina.

c) Coated with titania

d) Coated with alumina/ titania.
Table 7  Cyclic polarization data for coated and uncoated AISI 316L alloy in SBF solution at 37±1 ºC.

| Condition                  | $E_{\text{break}}$ | $E_{\text{Rep}}$ |
|----------------------------|--------------------|------------------|
| Uncoated ss316L alloy      | 800                | 500              |
| 50 nm of Titania           | 1350               | 1250             |
| 50 nm of Alumina           | 1350               | 1300             |
| 50 nm of Alumina/ Titania  | NO                 | NO               |

Figure 5( a & c) shows components of solution have precipitated on the surface of films, where alumina thin film has completed coverage and more adsorption (Figure 5 a) than that of titania thin film (Fig. 5 c), which seems less prevalent. The surface roughness of alumina and titania thin films increases after biocompatibility test. From EDX results (Figure 5 (b & d) it can be seen that the precipitates are found to be strongly enriched in calcium (Ca), approximately 16 % on alumina thin film and around 14.7 % on titania thin film. It means that the value of the calcium/phosphate (Ca/P) ratio is around 1.6 for alumina thin film and 1.47 for titania thin film which results good bonding to the bone during implantation because the ratio of Ca/P in the bone is 1.6 as reported by Qiyi-Zhang [27]. On the other side, alumina thin films have hydrophilic nature compared with the hydrophobic natural of titania thin films, which are because of high concentration of surface-exposed hydroxyl groups in alumina thin film deposited by ALD. The wetting properties of hydrophilic surfaces also have strong influence on the biocompatibility of alumina thin film [28]. Accordingly, titania hydrophobic surface showed a significant decrease of the biocompatibility. The biocompatibility of the alumina thin film deposited by ALD agrees with the results reported by researcher Dudley [29] and Muna et al. [30].
The bioactivity of alumina, titania and alumina/titania multilayers deposited on stainless steel 316L alloy with 50 nm thickness is investigated by cultured of Non-pathogenic Escherichia coli (E-coli) bacteria for 24 hr. at 37 °C as indications to the toxicity effects of thin films deposited. Thin film has less toxicity effects on non-pathogenic E-coli bacteria cultured means its bioactive film. The number of colonies living on the agar plate, after incubation at 37 °C for 24 h, was counted and studied according to equation (2). The results were expressed as mean colonies forming units per milliliter and antibacterial efficacy was determined on the basics of duplicates test result. The titania thin film has 15% reduction, alumina thin film has 20% reduction, and alumina/titania multilayers have 40% reduction. Another method of antibacterial is the diffusion zone as shown in figure 6. Where, the diffusion zone (number of bacteria died) is around 2 mm for titania thin film, 4 mm for alumina thin film and more than 6 mm for alumina/titania multilayers thin films respectively.

**Figure 5** SEM/EDX results of the biocompatibility test;

a) SEM image of alumina layer
b) EDX of alumina layer
c) SEM image of titania layer
d) EDX of titania layer
The small diffusion zone and less percentage of bacteria reductions mean that the titania thin film has less toxicity effects on E-coli bacteria used, which means more bioactive than that of alumina thin film and alumina/titania multilayers thin films.

![Diffusion Zone](image1)

**Figure. 6** Diffusion zone method of the bacterial cultured

4. **Conclusion**

The results showed that the good nanocoatings thickness control with excellent homogeneity and high area uniformity can be obtained from using ALD at 250°C stainless steel 316L specimens and Si-Wafer (100). The surface morphologies of the thin films have different nanoparticles size and shape depend on type of thin films deposited, Alumina or Titania. It was found that the alumina, titania and alumina / titania multilayer thin films deposited with 50 nm nanocoatings thicknesses by ALD at 250°C have great effect on improving the corrosion resistance of stainless steel 316L compared with the uncoated specimens electrochemically in SBF solution at 37±1°C. Alumina / Titania multi-layer thin films proved to be more corrosion resistant with respect to single film of alumina and titania at 50 nm thickness. While alumina has better corrosion resistance than titania. Biocompatibility of alumina film was more than that of titania film at 50 nm thickness, where more components of solution precipitated on alumina than that of titania according to determine of Ca/p ratio, which is equal to 1.6 for alumina and to 1.47 for titania. Bioactivity of titania film at 50 nm thickness with non-pathogenic E-coli bacteria was more than that of alumina and alumina/titania multilayers thin films, which means less toxicity of titania thin film deposited.

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