A Study The Bio Inert Ceramic Coated by Electrophoretic Deposition on Corrosion behavior of SS316L Alloy Used for Surgical Implant Applications

Murtdha Adhab siyah¹, Saba Abdulzahra Alshiaa²

³Sabea Amnas

¹Directorate of treatment and management of radioactive waste, Ministry of Science and Technology, Baghdad, Iraq, E-mail: saaabd@yahoo.com

²College of Science, University of Babylon, E-mail: saaabd@yahoo.com

³Directorate of treatment and management of radioactive waste, Ministry of Science and Technology, Baghdad, Iraq, E-mail: saaabd@yahoo.com

Abstract

Biocompatibility for the alloys which are used in the human body such as biomaterials depending on corrosion behavior. This study aim at research was to study the corrosion action and the biocompatibility of the samples of SS316L alloy uncoated and coated and compare the effect of coatings on corrosion behavior. Partially Stabilized Zirconia (PSZ) coating was do it by (EPD) technique on SS316L alloy. Structural investigation by using techniques including XRD, microstructure and crystalline of the coatings investigation in this study. Electrochemical measurement when effort the exams has been done in simulated body solution (Ringer and artificial saliva) in order to know the limitation and comparison the corrosion behavior of the gilder samples and clumsily samples as an evidence biocompatibility. Corrosion behavior for these coated specimens was more nobler than for the uncoated specimens.

Keywords: Partially Stabilized Zirconia, corrosion, electrophoretic deposition, biocompatible.
1-Introduction

Electrophoretic Painting (EPD) was discovered in 1809 by Reuss of Moscow. Many processes based on EPD Deposition have been described, including deposition of thick films, laminates, and body shaping. Significant interest has recently been focused on cathodic electrodeposition, which offers important advantages for various applications like cathodic electrolytic deposition which is a new technique in ceramic processing that has been used to produce a variety of ceramic thin films [1]. (EPD) is an industrial process in which slimly particles suspended in a fluid medium migrate under the influence of an electrical field and are deposited on an positive electrode. (EPD) is able to synthesis coating have thickness in ranging from a few micrometers to a few centimeters. Two electrode-position process techniques have been modified for forming ceramic films: electrolysis precipitation, using electrolytes finding solutions and ions it will be on behalf of cargo carriers. (EPD) was used particles comment in the solution like the ceramic dried that must be precipitate in the media organic or aqueous-organic media. And This is another method it is used for high-weight molecular samples, difficult to getting by Ionian conversion, and for polymer special types [2] [3]. The use of (ZrO₂) Zirconium as such a biomaterial it was beginning at late the sixties, first article concerning biomedicine relating to the application and uses of zirconium(ZrO₂). It has been published in 1969 by Helmer and Driskell, while the first article discussion using of (ZrO₂) manufacturing the ball heads for a total of Hip Replacement (THR), which is the current the main application of this ceramic biomaterial, was susceptible by In the early stages from development [4]. A lot of the solid finding solutions like (ZrO₂-MgO, ZrO₂-CaO, and ZrO₂-Y₂O₃) they were investigation for uses to biomedical applications. But years that followed researches have more concentrate on yttria ceramics, describe by minute grained microorganisms known as ceramics, which minimum of needs as such planting to undergo surgery are now prescribed by the benchmark ISO 13356 [5] [6].

2-Method and Materials

The material used in this work was austenitic (type SS 316L). Chemical analysis of this material was carried out using (XRF), X-MET 3000 TX from oxford instruments, Eng-land in Ministry of Science and Technology (iraq). Table (1) shows the nominal and the analytical chemical compositions of SS 316L alloy used in this work. Surface resistance of alloy plays an important role in corrosion behavior, hence, it is needful to get regular surface and requires careful samples preparation.
The SS316L alloy specimens were cut out in dimensions of (10×10 mm) for electrochemical tests and (20×20 mm) and 2 mm thick for immersion test. Specimens of (10×10 mm) were cut out from materials (SS316L).

The shaped specimens were molded using fast cold material up to 20 cm thick leaving the topside of the specimen exposed. The mounted samples were allowed to set for 30 mints and attention it was occupied to make sure that the musty will be not have any cracks or bubbles in the form/ samples interface. Contact electrochemical studies, suitable provision was made on the other side for electrical contact. schematic of *diagram of the molded specimen for microstructure evaluation and electrochemical tests escalated specimens were ground with SiC research in sequence on 120, 180, 220, 320, 500, 800, 1000, and 1200 grit to get mirror surface. The specimens were polished using polish cloth and alpha alumina 0.3 µm and washed with distilled water. The polished specimens were degreased with acetone trichloroethylene and cleaned in the same solution. The degreased specimens were washed with deionized water, dried and kept in a dissector over a silica gel pad and used for microstructure evolution and electrochemical investigation.

Kroll’s reagent containing from 45ml of Glycerol, 15 ml of HNO₃ and 30 ml of HCl it has been used to scratch for surface under optical monitoring. The time of exposure for Kroll reagent was half minute [7] . A beaker have capacity 500 ml was prepared to used as the deposition cell. A D.C. power supply control source was used to provide the electric power needed in this research. The resistance in power units was controlled, to obtain the optimum current or voltage until the experimental work of the coating process. The predetermined quantities of coating samples and polymer (dispersant) was milled for 120 minutes in a beaker, and Acetone as solvent to obtain thick slurry. The milled suspension was allowed to stand for 60 minutes. (for settling down of any rough particles), and then decanted carefully in the electrophoretic cell. The electrode set-up connected to the power supply source. The (EPD) coating process was carried out for various time intervals (180 minutes) at a constant voltage of 30V. After electrophoretic deposition, the cathode sample on which deposition was made was carefully extracted. The green deposition was slowly dried in the air at room temperature , followed by heating the specimens at 80 °C on the oven . The weight of coating was measured by weighting the specimens before and after the deposition process . The deposited samples were then sintered in a heated with Pumping inert gas (argon ) on the furnace at 900°C for one hour to getting better adhesive coating for samples. The coating thickness was determined in micron state (Seidner -7940 Riedlingen - W - Germ).

| Element wt% | Fe | N | Mo | S | P | Si | Mn | Ni | Cr | C |
|-------------|----|---|----|---|---|----|----|----|----|----|
| Actual Value | Rem. | 0.10 Max | 2.3 | 0.63 Max | 0.0245 Max | 0.75 Max | 2.0 Max | 10-14 | 16-18 | 0.03 Max |
| Analytical | Rem. | 0.08 | 2.2 | 0.01 | 0.02 | 0.67 | 1.61 | 11.81 | 16.18 | 0.27 Max |

**Table (1) chemical analytical composition of SS 316L alloy.**
3.1-Microstructure evolution

The microstructural evolution was investigated by means of optical microscopy using (Nikon category 120, Japan optical microscope), supply with digital camera style DXM 1200 F. The micrographs it has been analyzed across Nikon ACT- version 2.62, 2000 software. The microstructure investigation of the material used (SS316L) alloy shows in Figure (1) and appear microorganism of this alloy consists of solid solution (γ) which has constitution of nickel and chromium as substantial components dissolved in the matrix. Microstructure description is also present in the ASTM standard [8]. This grade of austenitic SS 316L alloy contains (16 -18) % chromium element, a ferrite-stabilizing and sufficient austenite-stabilizing elements, such as nickel, nitrogen, carbon and manganese, to render austenite stable at temperature room.

![Figure 1: The microstructure of uncoated wrought SS 316L. A (500X), B (1000X).](image)

3.1.1 XRD of Bio ceramic Coating

Figure (2) shows the patterns XRD of SS 316L alloy coated with (PSZ) partially stabilized zirconia by (EPD) technique and treated with heating at 900°C for 1 hour with pumping (Argon) inert gas to get purity coating layers. Shows the XRD patterns sign to the surface of the samples good quality coated with PSZ. They have strongest pattern lines in the for coated samples are (111), (200), (002), (311) and at 2θ = 30.081°, 35.307, 49.900°, and 59.780°, respectively which is responsible for ZrO₂(JCPDS-ICDD files # 17-0923 & 42-1164). And the samples coating have another pattern showed in peaks at angle 2θ = 28.803°and 58.882°which is appear for the (241) Zr₃Y₄O₁₂ (JCPDS-ICDD file # 29-1389) and (111) Y₂O₃ (JCPDS-ICDD file # 44-0399) on the sequences.
Electrochemical Studies

The electrochemical cell used in this study was fabricated according to the ASTM standards G5-87 [9]. The electrochemical cell containing six-necked flask of 500 ml capacity and it was equipped with a platinum electrode, which acts as the auxiliary electrode. Two auxiliary electrodes were used in the experiments for supply unified current distribution between tow electrodes the auxiliary electrodes and works of pole. we used a standard a calomel pole (SCE) as such the signal pole for measuring the electrode potential for corrosion cell. A Luggin capillary was stay in such in a way that the working pole and adjusted the a bout distance 2mm between them avoid ohmic decline. The temperature of the corrosion cell was adjusted at $37 \pm 1^\circ$C using of thermostated and water basin to have simulated the human body temperature.

3.1.2 - Solutions

Ringer’s solution and artificial saliva (Modified Carter’s solution) were use of such elec- trocuted for the investigations which due to its composition it was given in Table 2 and 3 were also used to compare the effect of various metal ions in solution on the electrochem- ical behavior of coated and uncoated samples.
Table (2) the chemical formula of Ringer solution [10, 11].

| NO. | CONSTITUENT | WEIGHT (gm/l) |
|-----|-------------|---------------|
| 1   | NaCl        | 9.00          |
| 2   | KCl         | 0.43          |
| 3   | CaCl₂       | 0.24          |
| 4   | NaHCO₃      | 0.20          |

Table (3) Chemical composition of modified artificial Saliva [12].

| NO. | CONSTITUENT | WEIGHT (gm/l) |
|-----|-------------|---------------|
| 1   | NaCl        | 0.70          |
| 2   | KCl         | 1.20          |
| 3   | KSCN        | 0.33          |
| 4   | NaHCO₃      | 1.50          |
| 5   | Na₂HPO₄     | 0.26          |
| 6   | KH₂PO₄      | 0.20          |
| 7   | Urea        | 0.13          |
All corrosion probably measurements were contacted referring calomel electrode (SCE) saturated a counter pole made from platinum frustration and measured potentiostate and software (Mlab 200, Bank electronic) was used for conducting the corrosion experiments. When the test sample specimen attained a fixed voltage, when effort polarization it has begun from an initially voltage of 250 mV less than the open circuit possibility and the survey was continuous up to 250 mV above the open circuit voltage [10]. The sample was photo scan in the direction of positive at a wipe average of 1.6 mV/s also it was monitored with respect for the voltage [11] [12]. The electrochemical parameters determined from the polarization curves (Figures 3, 4, 5, 6) are given in Tables (4 and 5).

### Table (4) electrochemical parameters of corrosion behavior to the SS 316 L uncoated.

| Solution                  | E corr mV | I corr µA | Mpy  |
|---------------------------|-----------|-----------|------|
| Ringer                    | -320      | 0.645     | 0.270|
| Modified Carter’s solution| -133      | 1.69      | 0.709|

### Table (5) electrochemical parameters of corrosion behavior to the SS 316 L Coated with PSZ.

| Solution                  | E corr mV | I corr µA | Mpy  |
|---------------------------|-----------|-----------|------|
| Ringer                    | -192      | 0.142     | 0.059|
| Modified Carter’s solution| -260      | 0.303     | 0.127|
Figure 3: Tafel relevance of effort Polarization from SS316L in Ringer Solution.

Figure 4: Tafel relevance of effort Polarization from SS316L in Ringer Solution coated with PSZ.
Figure 5: Tafel relevance of effort Polarization from SS316L in artificial saliva.

Figure 6: Tafel relevance of effort Polarization from SS316L in artificial saliva coated with PSZ.
4- Results and Discussions

1- Electrophoretic deposition is an alternative coating technique to control the stoichiometry, thickness and uniformity of the coated.

2- PSZ coating layers by electrophoretic deposition technique proves as available used for improving the corrosion impedance of type SS 316 L alloy for enhancing the biocompatibility of the implant or surgical uses.

3- The electrochemical behavior of uncoated and PSZ coated SS 316L alloy was investigated in artificial body fluid solution (Ringer and artificial saliva) at 37 °C, corrosion current for all coated samples is more noble than that of the uncoated samples. And the coated layer not only the protection of SS316L alloy from chloride attack in solution but also the suppression of dissolution of SS316L alloy ions via.

References

[1] Z. I., “Zhitomirsky i.”

[2] G. O. Brandusa Gihan, Gabriela Jicmon.

[3] O. O. Biest and L. J, “Vandeperre annu electrophoretic deposition of materials,” Rev. Mater. Sci, vol. 29, pp. 29–327, 1999.

[4] D. J.-M. Christel P, Meunier A, “Biomechanical compatibility and design of ceramic implants for orthopaedic surgery. bioceramics: material characteristics versus in vivo behavior,” Ann NY Acad Sci, vol. 523, pp. 234–56, 1988.

[5] C. Jerome, “What future for zirconia as a biomaterial?” Biomaterials.

[6] C. Piconi and G. Maccauro, “Zirconia as a ceramic biomaterial,” Biomaterials, vol. 19, pp. 2101–2127, 1998.

[7] G. Petzo,”Petzo.”

[8] M. H. ASM, “Properties and selection irons and steel,” vol. 9, 1985, pp. 223–1971.

[9] “Annual book of astm standards,” 1987.

[10] K. L. James and J. B. Mater, Res, vol. 5, no. 267, 1971.

[11] A. T. Kuhn and P. T. Rae,”Synthetic environments for the testing of metallic biomaterials,” P. T. Lee, Ed. ASTM STP 970, P.E. Francis and T.S. Lee (Eds.) Philadelphia, 79, 1988.

[12] “Quezanda castillo,” Journal of corrosion, vol. 60, no. 6, 2004, www.magasymposium.