Synthesis, Characterization and Optimization of Electrophoretic Deposition (EPD) Parameters of YSZ Layer on Ti-6Al-4V Alloy substrate

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Abstract. The objective of present work is to study the effect of electrophoretic deposition (EPD) parameters (voltage, time and concentration) using DC current based on the thickness, porosity and coating characterizations of YSZ layer on Ti-6Al-4V alloy substrate. The Taguchi approach was used in order to determine the optimal conditions for EPD and different criteria were applied for the deposit of biochemical coatings. Yttria stabilized zirconia powder (YSZ) was deposited on Ti-6Al-4V alloy substrate by electrophoresis using ethanol as a solvent under DC, to improve the quality of alloy surface and meet the requirements of biological orthopedic application activity. Ethanol was used as solvent to precipitate chitosan and YSZ on the alloy substrate. The composition of the surface and the cross section of coatings have been described by this electrode-enabled cathodic deposition for characterization. Many tests and inspections; zeta potential, water contact angle, XRD, SEM and optical microscopes were used to characterize surface morphology of YSZ layer. Optimum conditions for deposition of YSZ layer were used at 40 volt, 2 min and 1 g / l for suspension being at room temperature. The contact angles values of coatings were changed between hydrophilic 67.489 ° C and 35.914 ° C. Suitable percentages of porosity with pores size of 56.601-83.505 µm were obtained.

1 Introduction
Titanium (Ti) and its alloys are commonly used for manufacturing dental implants owing to their high biocompatibility and corrosion resistance. These favorable properties for in vivo implantation are due to Ti ability to form a passive oxide film, i.e. TiO2, within seconds after exposure to oxygen that makes it bio inert [1]. Even if the passive oxide is broken, it can be rapidly regenerated in the presence of oxygen, leading to protection of the metal surface [2]. This passive layer is very stable during function even under demanding mechanical and chemical conditions, such as during mastication and exposure to fluids in the oral cavity, thus protecting the titanium surface from corrosion [3]. Titanium and its alloys are widely used in dentistry and orthopedics for their mechanical properties, high strength-to-weight ratio, high corrosion resistance, and exceptional biocompatibility. An important feature of Ti-based alloys is their air-induced passivation, which creates a protective and stable layer of titanium oxide on the surface [4]. The metal of Ti-6Al-4V alloy has high biocompatibility, corrosion resistance, low elastic modulus, and low density[5]. 8 wt.% Yttria-stabilized zirconia (8YSZ) has exhibiting low thermal conductivity [2,5,6],
superior mechanical properties and high thermal expansion coincident [7]. Ti6Al4V alloy commonly used for biomedical implants for example, artificial knee joints, dental implants, artificial hip joints, bone plates, and also implants for dental products, such as crowns, bridges, and dentures, which are mainly produced by the precision casting method. and part for orthodontic surgery, bone fixation material as nails, knee, shoulders, spine and wrist joint replacement parts for hip joint, such as screws, plates, housing devices for the pacemaker and artificial heart valve, … etc [6]. Ti6Al4V alloy with α +β phases, which suitably treated, have an exceptional mixture of strength and ductility. They usually are stronger than the α or the β alloy individually [7,8]. Ti6Al4V alloy has higher biocompatibility than AISI 316L and cobalt-based alloy. This is return to the superior corrosion resistance of the Ti alloy than the other types referred to formerly [9]. The biomaterial surface is a chief issue forms undesirable reactions with the body. The chemical compositions on the surface and its topography are supposed to be essential in implants contacting bone [10,11] Because of EPD a simple and low cost method therefore it is widely used as coating way [12-13]. One of the motivating EPD usage involve surface coating of metallic implant utilizing calcium phosphate (Cap) which is then simultaneously reduces the potentially harmful metal ions release and enhances the bioactivity [14-15]. However, EPD has several advantages over reported other coating techniques such as its accomplishment at room temperature [16] Titanium and its alloys have recently been found widespread use in orthopedic surgery and dental applications in that Ti-6Al-4V has typically been used as bone plates and screws of easily removable implants after treatment based on specific requirement [17].

High biocompatibility, chemical stability, good aesthetic characteristics, flexural strength and fracture toughness are essential for dental materials in order to allow an efficient restoration of the tooth appearance and functionality. Biomedical grade yttria stabilized zirconia (YSZ), has been intensely investigated for this purpose.YSZ possess high flexural strength, high toughness, chemical inertness, and corrosion resistance with biocompatibility in the oral cavity. They are chemically inert materials, allowing good cell adhesion compared to other dental ceramics [18-20]. There have been several methods (electrodeposition, electrochemical, metal-organic chemical vapour deposition, Micro-arc oxidation, Plasma spraying, and electrophoretic deposition (EPD) [21,22,23], etc.) are available for deposition of YSZ material for various applications. But, these methods required highly sophisticated equipment for depositing the ceramic materials. Moreover, uniformity of the coatings remains challenging. On the other hand, electrophoretic deposition (EPD) process required simple equipment and provides highly packed uniform coating from the alcoholic suspension. The advantages of EPD process includes rate of deposition can be controlled by varying applied voltage, low cost and coating process can be completed in a few minutes [24]. Zirconia (ZrO2) is a bio ceramic and is bio inert, which shows superior mechanical behavior and biocompatibility. Therefore, adding ZrO2 into HAP improves the interfacial bonding
strength of the Ti substrate and coating. [25]. On versions of zirconia from tetragonal to monoclinic phase induces the increase in its strength and fracture toughness [26]. As a type of metallic oxide, zirconia is chemically stable at room temperature. However, it is possible to react powdered ZrO2 with water, alcohol or other organic solvents such as acetyl acetone under certain conditions. Actually, the natural surface of YSZ or zirconia has two similar forms: The Zr(OH)4•nH2O (hydroxide zirconium) and ZrO2•nH2O (hydrous zirconia) due to exposure to moisture in the air [27]. It is thought that the non-bridging hydroxo groups –OH in the former induces a condensation reaction between zirconia powders and thereby leads to hard agglomeration. The washing of powder with alcohol was ineffective to eliminate this agglomeration but helpful to reduce hard agglomeration in hydrous zirconia powders by the formation of ethoxide. surface chemistry of YSZ not only influences the colloidal behavior of the powder in a liquid (e.g. agglomeration) but also possible affects the consolidation of the powder compacts [28]. The aim of this work is devoted to optimize the EPD parameters including (voltage, time and concentration of suspension) using DC current by using Taguchi method to realize the YSZ coating on the Ti6Al4V alloy substrate depending on thickness values and then characterization of coating layers to select optimum condition.

2. Experimental Work

2.1 Electrophoretic Deposition (EPD) Coating Process

The material used in this research was Ti-6Al-4V alloy as plate of thickness 5mm provided from (Company: Shanxi Joint industry co., ltd) which tested in Science and Technology Ministry by using analytical instruments model XEPOS and its name SPECTRO. Table 1 shows the chemical composition of Ti6Al4V alloy.

| Element wt% | Al | Si | Ti | V | Fe | Ni | Sum of Concentration |
|-------------|----|----|----|---|----|----|----------------------|
| Concentration % | 5.94 | 0.09 | 89.37 | 4.05 | 0.25 | 0.3 | 100% |

Alloy plate was cutting into samples by wire cut machine with slow cutting speed and flow rate also used cutting fluid to avoid the heating result from the cutting. The dimensions of the samples were 5mm thick, 20 mm length and 10mm width determine according to the required test. One face of the samples was ground by SiC emery papers with 400, 600,800 and 1000 - grit. Then they were cleaned in distilled water, and acetone ultrasonic bath for 15 minutes. Chitosan (medium molecular weight with a degree of deacetylation of about 85% soluble in 1% acetic acid with purity (> 98%) (purchased from Sigma Aldrich) was used as a binder on Ti6Al4V alloy substrate. The important step in the experimental procedure of EPD is to prepare the aqueous suspensions. The first step was dissolve 0.5g/L of chitosan using 1% acetic acid [21]. Then solvent was added with 5% distilled water and then Nano powder of YSZ was added. All
suspensions were deagglomerated by a magnetically stirred. It was followed by a high-energy sonicator (Ultrasonic Processor, MIXSONIX Incorporated N.Y, USA) for 30 min. pH meter with acetic acid was used to adjustment range of pH value 4 of solutions by using (pH-EC-TDS Meter Portugal). Zeta potential was measured for each solution to ensure its stability. The EPD cell used in this study consists of a beaker and two electrodes immersed in the suspension as schematic EPD system used in this study is shown in Fig. 1. The EPD consists of a beaker and two electrodes immersed in the suspension. The Ti-6Al-4V alloy and stainless steel 316L were cathode and anode respectively. The deposition process was take place through three deposition conditions for yttria stabilized zirconia (YSZ) of concentration (1, 2 and 3 g/L), periods of time (2, 4 and 6 min) and voltages (20, 40 and 60 volt) were taken at room temperature. Then the coated samples were dried by air.

2.2 Design of experimental (DOE)

Taguchi's approach is a statistical tool of design of experiments (DOE) by using (MATLAB PROGRAMMING). It was used to analyze data and for modeling the influences of different parameters or factors of EPD method on output whereas the inputs are described as factors while the outputs are described as response variables. This approach was used to determine optimal design parameters for performance and cost.

![Figure 1. EPD system](image)

The objective was to select the best combination of controlling parameters or variables so that the Taguchi method was strongest with respect to noisily factors. In this study L9 (3^3) orthogonal array were used in experimental work to deposit of YSZ layer. Table 2 shows the experiments design according to
Taguchi’s approach and Table 3 shows the parameters of orthogonal array for YSZ layer that used in design of experiments. The detailed methodology is demonstrated in the flowchart (Fig. 2).

![Flow chart represents Taguchi methodology.](image)

**Table 3.** Parameters of YSZ coating.

| Parameters       | Voltage, V | Time, min | Concentration C%g/L |
|------------------|------------|-----------|---------------------|
| Voltage, V       | 20         | 40        | 60                  |
| Time, min        | 2          | 4         | 6                   |
| Concentration C%g/L | 1          | 2         | 3                   |
Table 2. L9 ($3^3$) Orthogonal Array for EDP parameters of YSZ layer.

| Parameters | Voltage (v) | Time (min) | Concentration (g/L) |
|------------|-------------|------------|---------------------|
| 1          | 1           | 1          | 1                   |
| 2          | 1           | 2          | 2                   |
| 3          | 1           | 3          | 3                   |
| 4          | 2           | 1          | 2                   |
| 5          | 2           | 2          | 3                   |
| 6          | 2           | 3          | 1                   |
| 7          | 3           | 1          | 3                   |
| 8          | 3           | 2          | 1                   |
| 9          | 3           | 3          | 2                   |

2.3. Microstructure Characterization

X-ray diffraction analysis has been performed on Ti6Al4V alloy specimen to determine the existing phases. X-ray diffraction device used is (Lab X, XRD – 6000) with 40 Kv and 30 mA. Scanning speed 2° per minute was used. The range of the diffraction angle was (20 – 80 °). The thickness of coating and its surface morphology was examined by optical and scanning electron microscopes. The distribution of elements at selected points /areas was detected by EDS detector. Zeta potential was necessary to study and analyze the stability of the suspension. This is one of the important tests in the EPD to ensure obtaining a homogeneous solution and thus ensure homogeneous coating layer. The particles were firstly dispersed in the corresponding solvent with a solid concentration lower than 1 g/l to obtain reliable results by ultrasonic treatment for 15 min. The results were ensured by the exposure of the suspension to the zeta potential measurement.

3. Results and Discussion

3.1. Taguchi Results

After the suspension preparation for YSZ preparation the statistical approach of Taguchi was utilized to choose the optimum conditions for YSZ layer preparation on Ti6Al4V substrate. Since the YSZ layer preparation aims to gain the highest thickness of coating. Thus three values of voltage (V), time (t ) and concentration (C ) were selected from signal -to- noise ratio (S /N) as the best one as illustrated in the table 4 and fig.2 which were obtained according to Taguchi design (L9) for thickness measurement results for YSZ layer with coating thickness 26µm using DC current. Table 5 shows the optimum conditions of YSZ coating thickness by using EPD with DC current.
Table 4. Signal-to-noise (S/N) ratio.

| Sample No. | Voltage, V | Time, min | Concentration, g/L | Thickness, μm | S /N | MEAN1 |
|------------|------------|-----------|--------------------|---------------|------|-------|
| 1          | 20         | 2         | 1                  | 22            | 26.8711 | 22.3333 |
| 2          | 20         | 4         | 2                  | 23            | 26.3651 | 21.0000 |
| 3          | 20         | 6         | 3                  | 24            | 25.1018 | 19.0000 |
| 4          | 40         | 2         | 2                  | 28            | 26.8108 | 23.0000 |
| 5          | 40         | 4         | 3                  | 26            | 26.4249 | 21.6667 |
| 6          | 40         | 6         | 1                  | 25            | 26.8052 | 22.3333 |
| 7          | 60         | 2         | 3                  | 23            | 26.8305 | 22.0000 |
| 8          | 60         | 4         | 1                  | 19            | 25.9988 | 20.0000 |
| 9          | 60         | 6         | 2                  | 26            | 27.9449 | 25.0000 |

Figure 3. S/N ratio and means for thickness.

Table 5. Optimum conditions of YSZ coating thickness by using EPD with DC current.

| Thickness, μm | Voltage, V | Time, min | Concentration, g/L |
|---------------|------------|-----------|--------------------|
| 60            | 2          | 2         | 2                  |
| 40            | 2          | 2         | 2                  |

3.2. Porosity and Thickness Measurement

The cross section of coating and its topography was examined by using optical microscopy. The results of the examination are illustrated in the Fig.4. Indicates the highest thickness of YSZ coating layer was 28
µm with dense, continuous and homogenous layer when the used conditions were 2%g/L of YSZ, 2 min deposition time and 60 V applied voltage. The percentage amount of porosity for every sample can be evaluated by using image-J program as shown in Fig.5. The results in Table 6 explain that the coating was obtained by using the above parameters achieved minimum porosity value equal to 2.0%

Figure 4. show the YSZ coating layer on cross section of specimens.
### Table 6. Shows the results of porosity values by using Image-J program.

| Slice   | Total Area | Average Size | Area Fraction |
|---------|------------|--------------|---------------|
| 1-DC-YSZ  | 138861     | 57.601       | 3.974         |
| 2-DC-YSZ  | 140309     | 62.132       | 5.162         |
| 3-DC-YSZ  | 137013     | 70.668       | 3.454         |
| 4-DC-YSZ  | 138861     | 70.429       | 4.352         |
| 5-DC-YSZ  | 142259     | 72.947       | 2.010         |
| 6-DC-YSZ  | 135005     | 83.505       | 2.795         |
| 7-DC-YSZ  | 141810     | 51.059       | 4.209         |
| 8-DC-YSZ  | 133089     | 64.038       | 9.480         |
| 9-DC-YSZ  | 140700     | 70.764       | 6.181         |

**FIGURE 5.** Variation in porosity area fraction according to the YSZ coating process variables.
3.3. X-ray diffraction analysis

Fig. 6 shows the XRD analysis for Ti6Al4V alloy in as received condition. It explains that the structure is consisted of (α+β), α phases represents α- stabilizing element as Al element tend to stabilize α phase, while V element acted as β phase stabilizer [18].

![XRD analysis of the Ti6Al4V alloy](image)

Figure 6. XRD-analysis of the YSZ powder.

3.4. Solution stability

The measured of pH value of the suspension was pH4 which led to obtain Zeta potential with positive as depicted in the table 7, which enhance the homogeneity of YSZ precipitation. Increasing mobility by increasing absolute values of zeta potential. The high YSZ particles mobility is returned to the positive zeta potential value [2]. The zeta potential and mobility of YSZ nanoparticles for the suspension are illustrated Table 7 and Fig. 7 respectively.

| Material | Frequency | Frequency Shaft | Mobility | Zeta Potential |
|----------|-----------|-----------------|----------|----------------|
| YSZ 3-0  | 255.25    | 5.25            | 0.67     | 33.74          |
3.5. Contact Angle Measurement

The hydrophobicity or wet-ability of the coated Ti6Al4V alloy and uncoated samples are measured using a steady drop of distilled water by measuring the solid and liquid contact angle via optical contact angle equipment type CAM 110-O4W which is provided with CCD camera. The coating lead to decrease the contact angle from 67.489º to 26.755º which in turn converted the uncoated surface of the Ti6Al4V alloy from hydrophobic to hydrophilic. Surface coating was more hydrophilic in the YSZ coating layer. In this case the bone regeneration will be more easy and rapt. When there are decreasing in contact angle with YSZ coating is deposited on substrate coating. This result is agreement with reference [17]. The above description of the contact angle values for uncoated and coated Ti7AI4V alloy substrate are shown in the fig. 8.

Figure 7. Zeta Potential and Mobility of YSZ.

(a) Zeta potential                         (b) Mobility

Figure 8. Water contact angle measurements of (a) Uncoated and (b) YSZ-coated titanium samples.
3.6. Microstructure with EDS analysis

The microstructure of coated Ti6Al4V alloy observed by SEM with using EDS analysis were shown in the fig.9 and 10 respectively. Fig.9-a shows the nearly homogeneity of coating YSZ on the alloy surface while the Fig.8-b shows YSZ coating thickness which is approximately equal to 25µm. Fig.10 a & b indicates the EDS elemental analysis of uncoated Ti6Al4V alloy substrate and coated Ti6Al4V alloy with YSZ layer respectively. This chemical composition is in well agreement with the composition of the used alloy.

![Figure 9. shows the SEM images a). Surface morphology and b). Coating thickness of YSZ](image1)

![Figure 10. shows the SEM and EDS of (a) uncoated Ti6Al4 substrate (b) Coating of YSZ](image2)

4. Conclusions

1- The optimum conditions for EPD of YSZ coatings on Ti6Al4V alloy substrate depending on the roughness and thickness values are, 40V, 2 min, and 2 g/L.
2- XRD analysis proved that EPD technique was suitable for Ti6Al4V alloy due to the existence of phase YSZ coating.
3- Good stability was obtained with all solutions for deposition coatings which confirmed by zeta potential. The higher stability value of solution was found 33.74 for 100% YSZ + 0.5% chitosan.

4- Water contact angles measurements for samples showed that the YSZ coating surfaces are altered between 67.489º which considered as a hydrophilic to 26.755º which considered as super a hydrophilic.

5- From microstructural observations, it was seen that the YSZ layer is homogeneous, dense, and continuous coating layer on alloy substrate.

6- Suitable percentages of porosity with pores size of 56.601-83.505 µm were obtained.

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