Electrophoretic deposition technique for coating nano “TiO2” on SS316L alloy

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ABSTRACT

The aim of this work was to use the electrophoretic deposition (EPD) technique for the deposition of nano-”TiO2” layers directly on an SS316L substrate. The nano-”TiO2” films were deposited on an SS316L alloy with various parameters, such as potential, time of deposition and annealed temperature—the optimum parameters required for the development of stable nano-”TiO2” layer coating. The results obtained from the X-ray diffraction (XRD) and FESEM tests in this study showed that the “TiO2” layers sintered at 400 °C were amorphous. The XRD patterns showed the anatase phase at 600 °C for 1 h. The heat treatment of “TiO2” layers led to good adhesion with the SS316L alloy. The heat treatment above 500 °C led to the agglomeration of nano-”TiO2” layers from 28 nm to 78 nm at 600 °C. The thickness of the nano-”TiO2” coating increased with the deposition time and potential. The optimum thickness of the nano-”TiO2” coating on the SS316L alloy was 50 µm at 5 min and 30 V. The increase in coating thickness with the time and potential of deposition led to problems, including increases in roughness and weak adhesion of the coating layer.

1-Introduction

Organic colorants have become an inevitable part of our life. Synthetic dyes have replaced natural types in paper, clothes, drugs, and even food industries. However, the waste of these materials is considered as pollutant and dye treatment has become a scientific challenge. The membranes, adsorbent, floated foams, and coagulation agent are usually applied to remove the pollutants. These methods are not efficient and exert side effects. Oxidation of pollutants is a modern method with high efficiency and without any destructive effect. A method entitled “*advanced oxidation process (AOP)*” has been developed. The photocatalytic reactions provide the required conditions of AOP; therefore, the semiconductor materials are identified as suitable agents for AOP [1].

Cu due to its unique properties, including attractive colors, excellent electrical and thermal conductivity and ductility in cold and heat, is widely used in various industries [1]. But an important problem with the use of Cu equipment is the low wear resistance of this metal [2]. On the other hand, in many applications, such as marine industries, higher corrosion resistance is Surface modification of metallic alloys with ceramic coatings gained unanimous attention from the industry and researchers, as it produces properties such as erosion, wear, corrosion and oxidation resistances. However, the coating is only useful when it is stable and the layer coating and substrate have good adhesion[1].

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Electrophoretic techniques started at the beginning of the twentieth century [2] In 1917, Davey was given a patent for his clear for electrophoretic picture. In the past 40 years, the electrophoretic deposition (EPD) process was developed and has been widely used in ceramic technology [3, 4]. EPD is generally used for novel applications in the treatment of progressive ceramic materials and coatings. It has just become the interest of laboratories, researchers and industry fields because of its benefits, such as applicability to large areas, uncomplicatedness in setup, easy coating into conductive substrates, good regulation of coating layers, short development time and low-cost equipment [5-7] In this study, EPD and the factors affected by EPD, stability of suspension, use of EPD for the deposition micro and nano sizes and applications of EPD were presented [8-10]. EPD is a two-phase route: charging of the particles in the suspension and travelling of the charged particles in the direction of an electrode. Some agents, such as water, to the electrolyte can be added to control the packing density of the particles with an adjustment in current. EPD continues to gain interest for its responsiveness for the deposition of constant coatings[7, 11] To develop the wear and corrosion resistance of the alloys, some studies concentrated on the development of thin films [12-15] EPD is a colloidal processing technique for the deposition of materials with different particle sizes—from nano to micro—suspended in solution upon the presentation of an outer electric media. It is an increasingly developed process for engineered materials because of its low cost, stability and efficiency[16, 17]. Jason Bandy et al. used EPD to deposit nano-"TiO2" for solar-cell applications and showed that EPD can be used to control the porosity by controlling the electric potential and thus the current. An increase in current leads to more evolution of hydrogen gas, resulting in more porosity in the coating layers. The coating thickness is also tuneable by changing the deposition time [9]. Niazi et al in 2015 [18] presented the "TiO2"–Al_2O_3 coatings by using EPD in different potentials. At a high potential, the changes between the values of micro hardness are very apparent due to the incorporation of more alumina nanoparticles at high voltages. The micro hardness increased with the increased voltage. The hardness of "TiO2"–ZrO_2 increased with the increased value of ZrO_2 C. T. Kwok et al. investigated the hydroxyapatite coating by using EPD on Ti-6Al-4V and the effect of the coating layers on the corrosion behaviour of the Ti-6Al-4V alloy [1, 19]. The use of EPD in the biomaterials field possibly started with the progress of hydroxyapatite coatings on Ti alloys in 1986 [20]. The applications of EPD in the biomedical field are starting a variety of functional, nanostructured and composite coatings; layered and functionally classified biomaterials; porous biomaterials; tissue scaffolds; thin films; drug delivery systems and biosensors; and deposition of biopolymers, bioactive nanoparticles, carbon nanotubes and biological entities in progressive nanostructured biomaterials. In the field of biomaterials, EPD can be a choice for the development of advanced biomaterial (nano)structures as coating on biomaterial alloys [21]. "TiO2" layers are very important materials with numerous applications, such as self-cleaning glazing [22], water purification [23], sensors [24], photo-catalyst and photovoltaics[25, 26], corrosion-protective coatings and coatings for biomedical applications [27]. In this study, "TiO2" coated on SS316L was used for biomedical applications. The SS316L alloy exhibited increased corrosion resistance, and the best surface of the SS316L alloy that is biocompatible with the human body was found.

2. Experimental work

2.1 Preparation of suspensions

A suspension for EPD is a complex structure in which each factor has an important effect in changing the quality and type of deposition. The suspensions were prepared by adding 10 g "TiO2" powder to 100 ml of absolute ethanol purchased from Merck Chemicals without further purification, where 99.7% of the particles had dimensions less than 25 nm. The suspension of 0.22 g polyvinyl butyral dissolved in ethanol was used as a binder. A binder is important to increase the adherence power of the deposited particles of material and prevent cracking in the coating surface. Moreover, 0.25 g phosphate ester was used as a dispersing agent for the suspension. The phosphate ester must be added before the polyvinyl butyral to avoid competitive adsorption [28]. The suspension was agitated for 300 s and placed in an ultrasonic bath for 15 min. Subsequently, the suspension was mixed using a magnetic stirrer.

2.2 Substrate preparation

Rod-type SS316L was used in this study. It was cut into circular samples with a diameter of 20 mm and thickness of 4 mm. These samples were ground with silicon carbide (SiC) papers from 120 grit to 1200 grit and subsequently polished using diamond pastes to obtain the mirror surface finish. The SS316L samples were cleaned thoroughly with distilled water, washed and ultrasonically degreased with acetone and dried before the coating process [29] .

2.3 EPD coating

To conduct the EPD experiments, an aluminium thin sheet was cut into 20 × 20 mm^2 and used as cathode electrode, and the SS316L alloy samples were used as the anode. Each electrode was placed between the wood slid to ensure a constant distance of 1 cm. The parameters, such as time (3 min), voltage (30 V) and thickness of the “TiO2” layers (50 µm), were selected to control the “TiO2” layer thickness and achieve the best coating layers with stability and homogeneity. The coating layer with a thickness above 50 µm exhibited more roughness and low adhesion between the coating layers and SS316L substrate. A constant voltage of 30 V was applied for EPD with a constant current density of 0.1 mA/cm^2 for the different times at room temperature, as shown in Figure 1. To prevent the agglomeration of the “TiO2” particles in the suspension, the suspension was treated in an ultrasonic bath for 10 min. Subsequently, the “TiO2” suspension was placed on a magnetic stirrer to get the best condition suspension for the deposition process. The “TiO2” coating was annealed at 400 and 600 °C for 1 h at 3.5 °C/min in furnace to evaporate any unstable components.

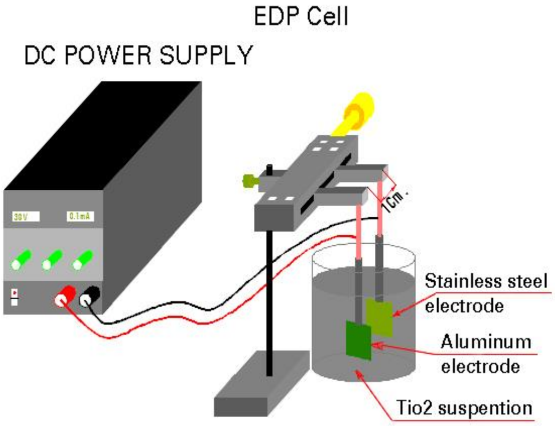

Fig.1. Presented electrophoretic deposition cell used in this study

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3. Result and discussion

3.1 XRD

“TiO2” has different phases, which are very sensitive to annealing temperature. At temperatures lower than 400 °C, the “TiO2” film is amorphous [30] “TiO2” layers prepared using electrodeposition are amorphous up to 350 °C [31, 32]. Figure 2 shows the XRD patterns of the SS316L alloy coatings with “TiO2” by EPD at different heat treatments of 400°C and 600 °C. The XRD pattern of the SS316L substrate is in Figure 2. The XRD spectrum of sample coated with “TiO2” at 400 °C exhibited an amorphous phase of “TiO2”. Only the peaks that came from the SS316L alloy (substrate) were observed. When the heat treatment increased to 600 °C, anatase peaks were observed strongly in the pattern located at $2\theta = 25.291°$, 38.580° and 48.05° corresponding to the (101), (112) and (200) crystal faces, respectively (JCPDS PDF number 21-1272). The SS316L phase located at $2\theta = 44.289°$, 51.540° and 75.901° corresponded to the (111), (200) and (220), respectively (JCPDS PDF number 38-0419). Crystal faces was still observed in the XRD spectrum.

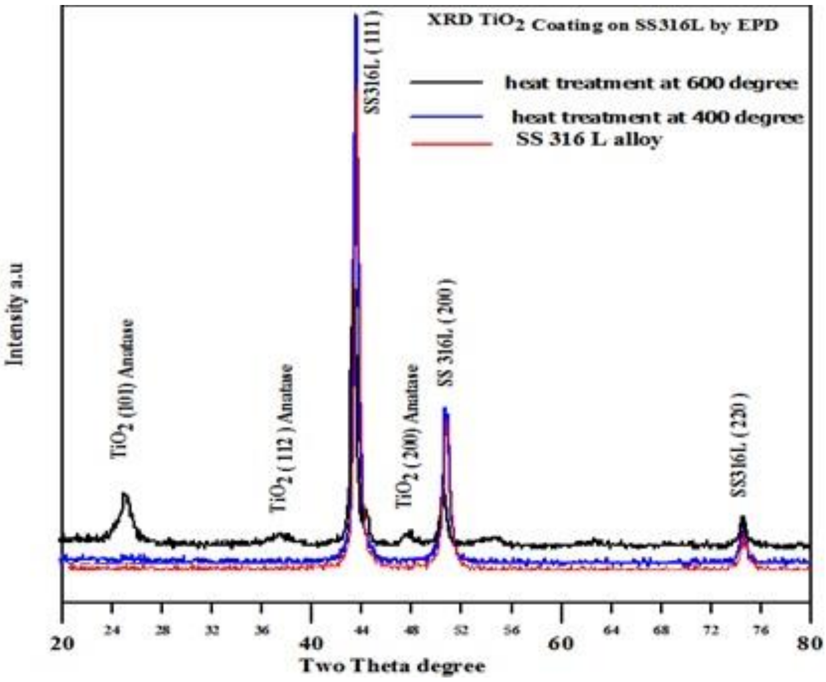

Fig.2. XRD of "TiO2" coated on SS316L substrate by electrophoretic deposition.

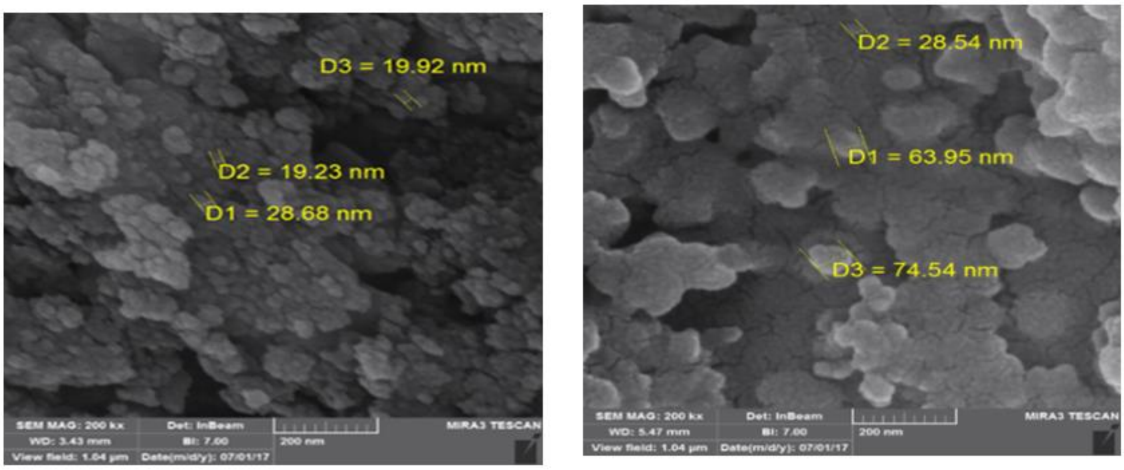

Fig.3. FESEM images presented particle size of "TiO2" films prepared by electrophoretic deposition on SS316L with heat treatment at 400 ^{O}C and (b) with heat treatment at 600 ^{O}C. D1, D2 and D3 the values of particle size

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3.2 Surface morphology of the "TiO2" layers

FESEM was used to characterise the morphological structure of the "TiO2" layers. FESEM images showed that the average values of grain size of anatase "TiO2" films increased with heat treatment. The high-heat treatment led to the agglomeration of the nano-"TiO2" coating from 28.68 nm at 400 °C to 74.54 nm at 600 °C (Figure 3a and 3b). The nano-"TiO2" coating prepared using EPD was successfully applied to SS316L substrates. The FESEM images and EDX investigation showed that the EPD coating technique can be used to get high-purity and nanosize layers. The FESEM images are shown in Figures 4. The anatase "TiO2" coating and the corresponding EDS are shown in Figures 4b and 4d. The peaks of titanium in the spectrum were due to the coating by EPD (Fe, Cr and Ni). The peaks in the spectrum resulted from SS316L substrate, and the Au peak in the spectrum was due to the preparation process of the samples for FESEM analysis.

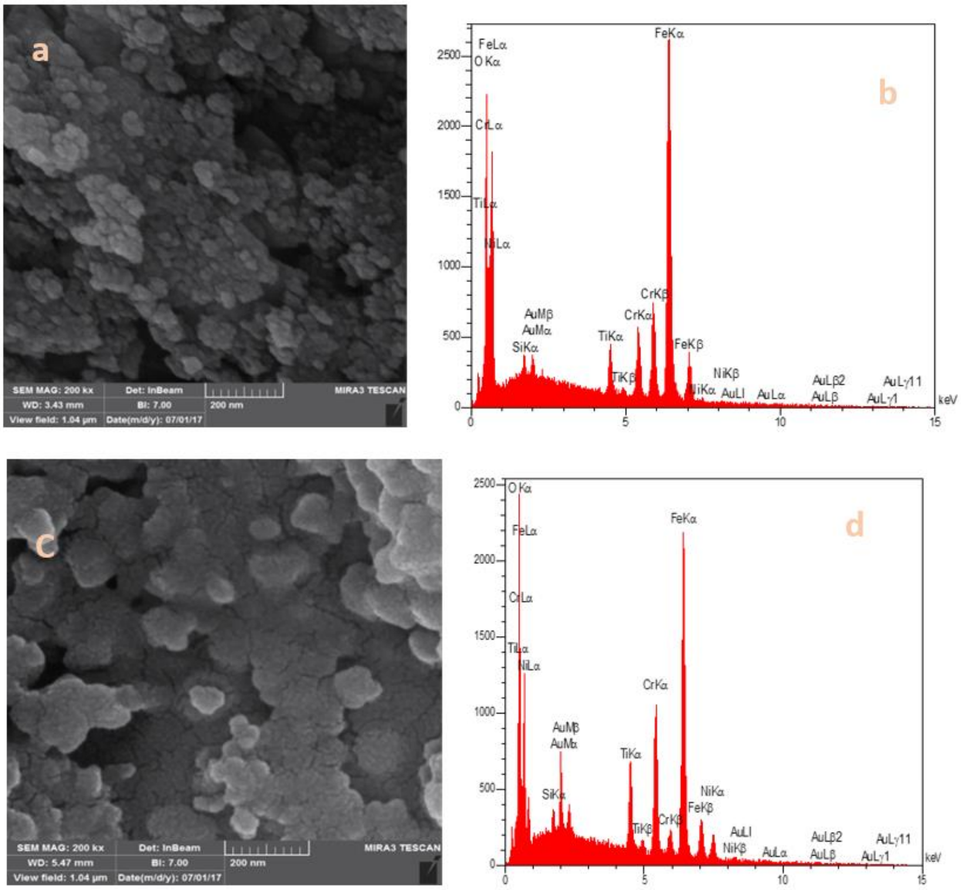

Fig .4. a: FESEM image and 4b: Energy Dispersive X-Ray Analysis (EDX) for “TiO2” coating on SS316L after heat treatment at 400^{O}C. c: FESEM image and d: Energy Dispersive X-Ray Analysis (EDX) for “TiO2” coating on SS316L after heat treatment at 600^{O}C.

Table .1. EDX Quantitative Results for “TiO2” coating on SS316L after heat treatment at 400^{O}C.

Elements	Line	Int	W%	A%
C	Ka	21.2	4.00	10.49
O	Ka	231.5	24.03	47.30
Si	Ka	18.1	1.60	1.80
Ti	Ka	64.1	4.60	3.03
Cr	Ka	115.2	8.22	4.98
Fe	Ka	668.0	57.25	32.28
Ni	Ka	2.2	0.20	0.11
Au	La	0.6	0.10	0.02
			100.00	100.00

Table .2. EDX Quantitative Results for “TiO2” coating on SS316L after heat treatment at 600^{O}C.

Elt	Line	Int	W%	A%
C	Ka	21.4	3.45	8.92
O	Ka	257.2	25.71	49.81
Si	Ka	17.0	1.31	1.45
Ti	Ka	121.2	7.74	5.01
Cr	Ka	237.0	15.79	9.41
Fe	Ka	552.8	42.25	23.45
Ni	Ka	44.8	3.66	1.93
Au	La	0.6	0.08	0.01
			100.00	100.00

3.3 Effect of coating thickness on the “TiO2” layers

The “TiO2” film thickness deposited at 1 min to 3 min with a constant of 30 V was found to be 15.64, 23.89 and 33.69 µm at 6, 9 and 12 min

(63.97, 96.79 and 102.41 µm), respectively (Figure 5 a, b, c and d). Surface roughness increased with the increases of “TiO2” coating thickness (Figure 5 a, b, c and d). The “TiO2” layer deposited on the SS316L alloy showed cracking in the surface of the coating layer, and the coating exhibited weak adhesion to the substrate (Figure 5d) and could see in figure 5 d the coating was be formed as a multilayer of “TiO2” and the outer layer has weak adhesion with the substrate and become separated that make it has low mechanical properties and could not protect the substrate SS316L from corrosion and it is destroyed with any pressure of low mechanical forces.

4.Conclusions

Titanium oxide films were prepared in the laboratory by using EPD at a cost and with simple equipment. The EPD was very successful in depositing nanomaterials. The stability of the coating film preparation by using EPD depended on the experimental deposition parameters, such as potential, time of deposition and the thickness of coating, which were very important for obtaining stable coating layers. The increase of deposition time increased the thickness of the coating and, by using EPD, produced thick films. However, above the thickness of 63 µm, the “TiO2” films exhibited roughness at 96 µm, and the “TiO2” layers showed cracking at 102 µm. The particle size was affected by the heat treatment. The “TiO2” aggregated and the particle size increased with increasing heat treatment.” The XRD analysis of “TiO2” coatings showed principally anatase phases at 600 °C. And could see in figure 5 b the coating was be formed as a multilayer of TiO2 and the outer layer has weak adhesion with the substrate “and become separated that make it has low mechanical properties and could not protect the substrate SS316L from corrosion and it is destroyed with any pressure of low mechanical forces.

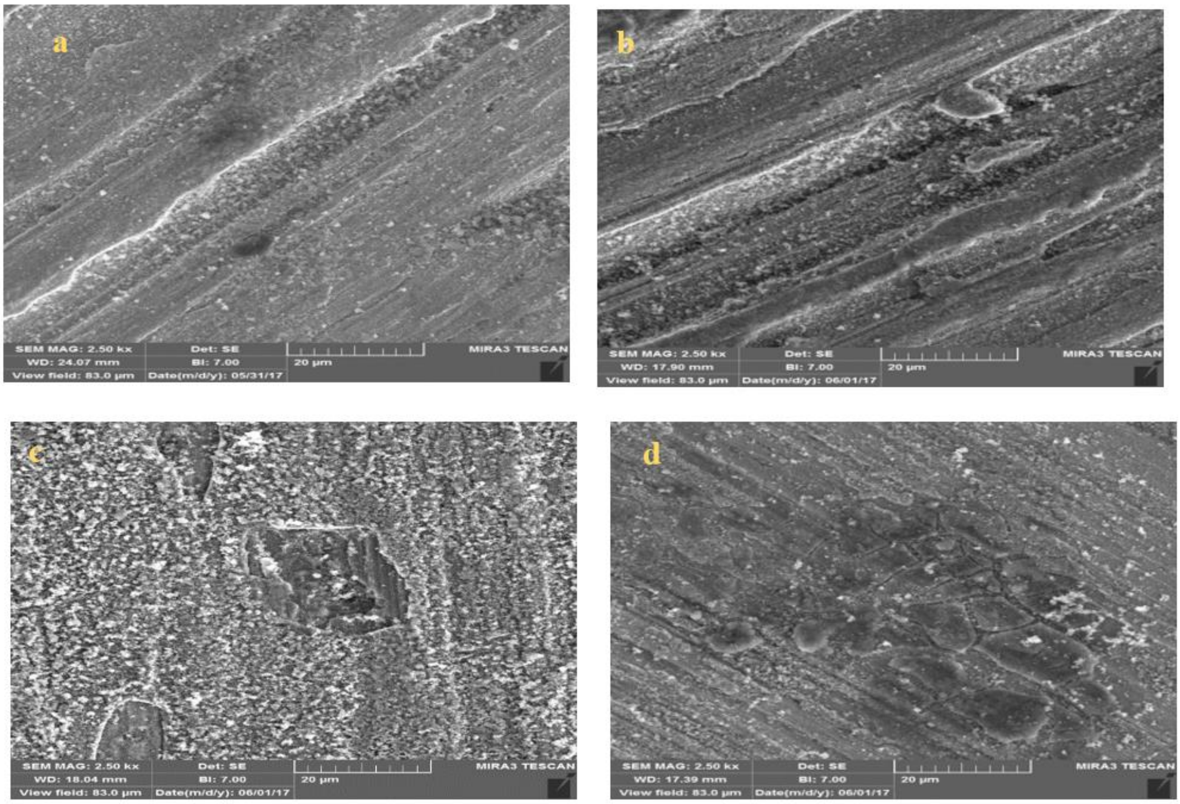

Fig .5. FESEM images presented the roughness increased of surface with increased the thickness of coating.

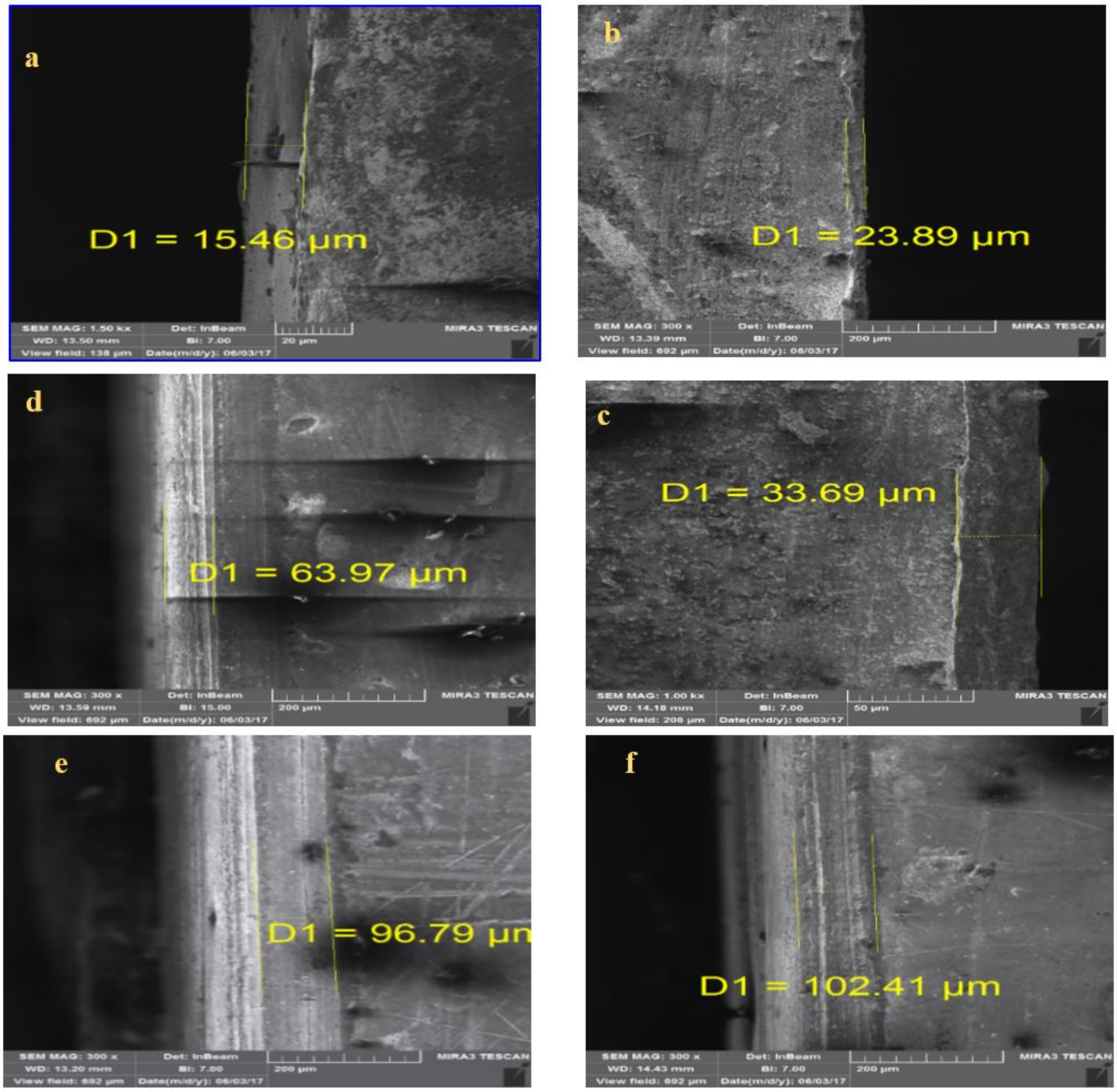

Fig.6. Cross section of “TiO2” coating by EPD with different time.

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