

Effect of Voltage and Deposition Time on the Electrophoretic Deposition of Hydroxyapatite Coatings

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ABSTRACT

The stainless steel 316L has been successfully coated by hydroxyapatite (HA) for increased their biocompatibility. The purposes of this research are to investigate the effect of applied voltage and deposition time on the properties of HA coatings. Stainless steel 316L with size of 20x20x1 mm³ put into alcohol and water solution for 1 hour. HA solution was prepared in ethanol solution used magnetic stirrer for 2 hours and 200 rpm. Subsequently the HA powder was deposited by electrophoretic deposition (EPD) using carbon as anode at 40, 60 and 80 volt and deposition time 2, 7, 12 and 17 minutes. It was observed from SEM analysis that HA deposited on stainless steel increased with higher voltage, thickness of HA layer on stainless steel was 30, 45 and 55 μm for deposition 17 minutes and applied voltage 40, 60 and 80 volt. Thickness of HA layer on substrate increased, as the time deposition increased with result 30, 35, 50 and 55 μm at deposition 80 volt and deposition time 2, 7, 12 and 17 minutes. The intensity of HA peaks in the coated substrate was highest at 80 volt and deposition 17 minutes. HA coatings at applied voltage 80 volt and deposition time 17 minutes was obtained suitable for bone plate.

KEYWORDS: Bone Plate, Coating, Electrophoretic Deposition, Hydroxyapatite

1.0 INTRODUCTION

Bone as functional tissue in the human body has various roles, such as skeleton, protecting many internal organs, and interconnection between muscles so enabling movements in human activity[1]. Accident and sickness causing of damaged bone. Damage bone cause interference of movement on human body. That damage can be resolved using implantation to fixation of fracture bone to real position (reposition) and maintained that position while healing time (immobilization).

For bone material implants commonly using as Stainless steel (SS), Co-Cr and Titanium (Ti) Alloy. SS has been widely used by orthopedic implant material because of its mechanical strength and the capability to bend and shape the implant, low cost, non-toxic, high corrosion resistance and low impurities but cannot affect bioactivity on human body [2]. Hydroxyapatite (HA) coating is preferred to be one of methods to prevent the problem of stainless steel. Hydroxyapatite (Ca₁₀(PO₄)₆(OH)₂) is well established as a biocompatible material of forming a good bond with natural bone. HA coated stainless steel implants integrate the bioactivity of HA and provides protection to substrates against corrosion and load bearing capability of the implants.

Many techniques have been used to deposit HA, such as dip coating, thermal spraying, physical vapor deposition (PVD) and electrophoretic deposition (EPD).

EPD is a deposition method using separation component or charge particles based on different migration in electric field. The mechanism of electrophoretic deposition involves two steps. The first step, electrophoretic is when an electric field is applied between two electrodes and charged particles suspended in a suitable liquid move toward the oppositely charged electrode. The next step is deposition, the particles accumulated at the deposition electrode and create a relatively compact and homogeneous film [3]. EPD has advantages of needs simple apparatus, not need high temperature, low cost and not need transformation phase when deposition and strong deposition [4].

2.0 MATERIALS AND METHODS

HA powder used in this work was synthetic HA prepared from sea shell using low temperature hydrothermal method. Stainless steel 316L was used as substrate, carbon as positive electrode, and ethanol as suspense.

The electrophoretic deposition on 316L stainless steel was determined at 40, 50, 60, 70 and 80 volt at a constant time of 2, 7, 12 and 17 minutes. This experimental was in three stages, first stage is preparation of stainless steel, cut into 20x20x1 mm dimension. The sample was then polished by 500 grid of sand paper. Then sample put into alcohol and water solution for 1 hour.

The second stage is EPD process, HA solution was prepared in ethanol solution with the help of magnetic stirrer. Carbon used as the cathode and 316L as the anode. SS coatings were further characterized using reflection mode of Fourier Transform Infrared (FTIR), phase identification was carried out directly using X-ray diffractometer (XRD) and cross-section examination of coating was performed by Scanning electron microscopy (SEM).

3.0 RESULTS AND DISCUSSION

3.1 SEM Analysis

Figure 1 shows the SEM observation using 500x magnification. Samples with the applied voltage 40, 60 and 80 volt has thickness around 30, 45 and 55 μm . Sample with higher voltage has larger thickness than other samples, this is caused HA powders can be deposited quickly if greater applied voltages are used.

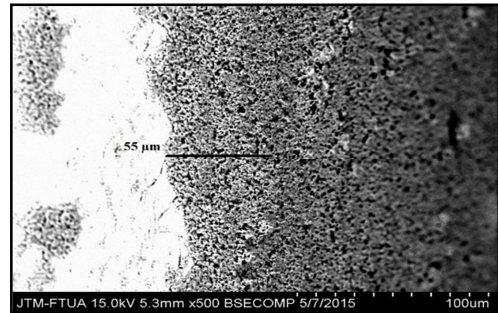
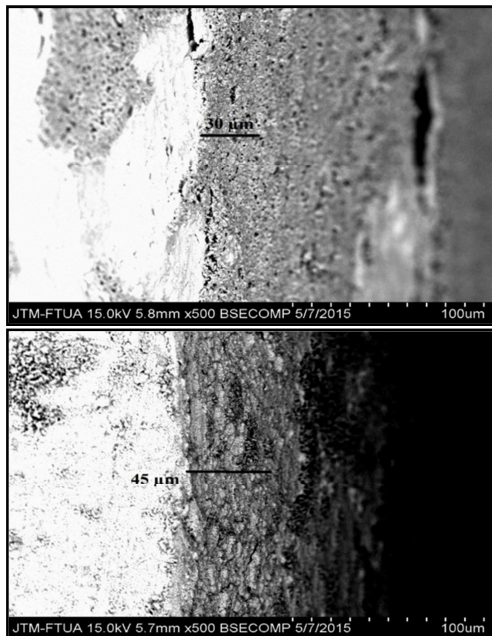


Figure 1: Cross-sectional SEM results for sample with applied voltage of (a) 40 volt (b) 60 volt and (c) 80 volt

HA thickness increases with increased deposition time, for a fixed electric field when the deposition time increases the deposition rate decreases. Figure 2 shows the thickness of HA with variation of the deposition time. From cross-sectional SEM result, for sample with deposition time 2, 7 and 12 minutes has thickness around 30, 35 and 50 μm .

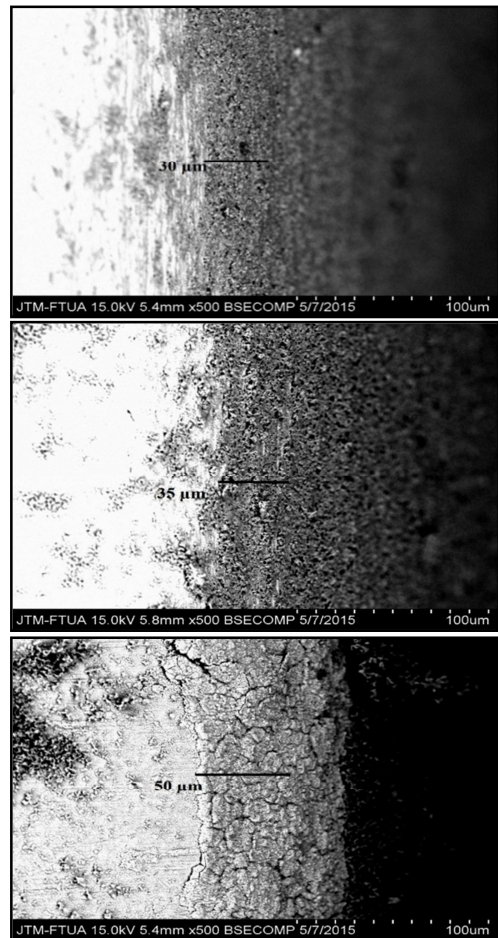


Figure 2: SEM result for HA coating with deposition time of (a) 2 min (b) 7 min (c) 12 min

Figure 3 shows the morphology and size of the HA coating on 316L SS, from the figures it is observed that HA is uniformly deposited on the surface. Grain size of the 80 volt (a) sample look smaller than the 40 volt (b) sample. For sample with deposition time variation, the 17 minutes sample has more the presence of HA powder on 316L surface compare than the 2 minutes sample. This is because growth of the HA crystal occurs with greater deposition times and the crystal cover up maximum area on the implant surface leaving very small holes.

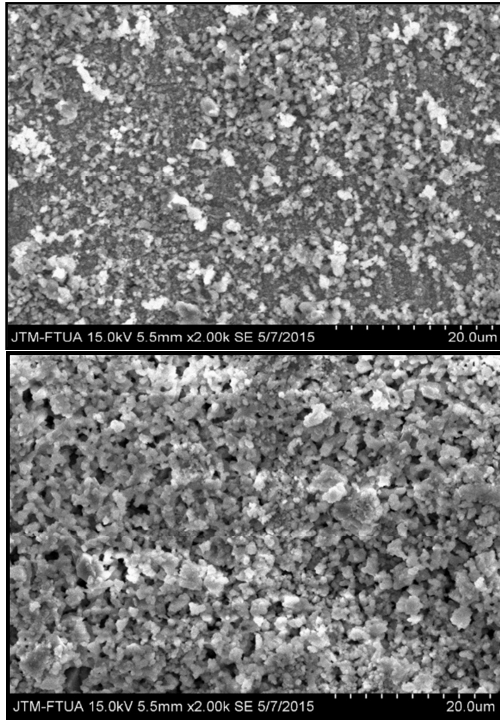


Figure 3: SEM images for HA coating (a) 2 min (b) 17 min

3.2 XRD Analysis

Figure 4 shows the X-ray diffraction patterns of HA powder that was coated on 316L SS samples for 40 and 80 volt. From the analysis of XRD data obtained that the peaks are match with the standard peaks of HA in the JCPDS No. 09-432 with hkl (002), (211), (112) and (300) at 25,879°, 31,773°, 32,196° and 32,902°. Peak of the 40 volt sample at 25,82°, 31,74°, 32,13° and 32,82°. In the 80 volt sample at 2θ 25,82°, 31,74°, 32,13° and 32,82°.

For sample with different deposition time, HA peak increased with increased deposition time. Figure 5, peak of 2 minutes sample at 25,82°, 31,74°, 32,13° and 32,92° but has lower peak than 17 minutes deposition sample. The highest peak of 2 minutes sample is Fe, this is because only SS 316 coating by small amount of HA.

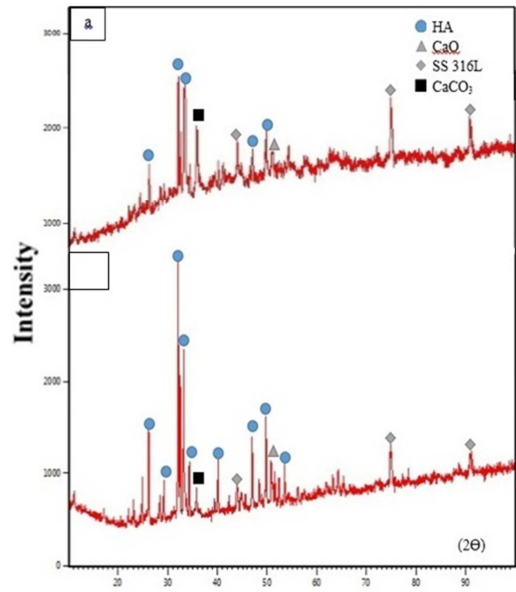


Figure 4: XRD pattern for HA coating at different applied voltage

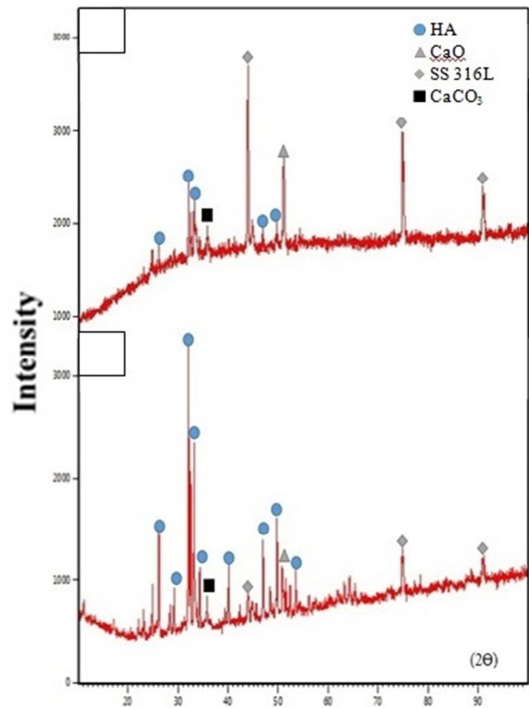


Figure 5: XRD result for coating with deposition time

3.3 FTIR Analysis

FTIR identify the functional groups in the sample. Functional groups that are identified PO_4^{3-} and OH^- in the range 4000 – 600 cm^{-1} . PO_4^{3-} groups appear at wavelengths from 1200-1100, 958, 962 and 605 cm^{-1} and OH^- groups at 3700-2500, 667, 630 cm^{-1} . Figure 5 show the FTIR spectra of the coating at different using

voltage. In 40 volt sample PO4 ribbon appear at 1185 and 942 cm^{-1} , when OH- groups appear at 643 cm^{-1} . PO4 group in 80 volt sample appears at wavelength 1194 and 934 cm^{-1} , when OH- group appear at 651 cm^{-1} . The results obtained spectrum for each researcher of HA is different but still in adjacent value.

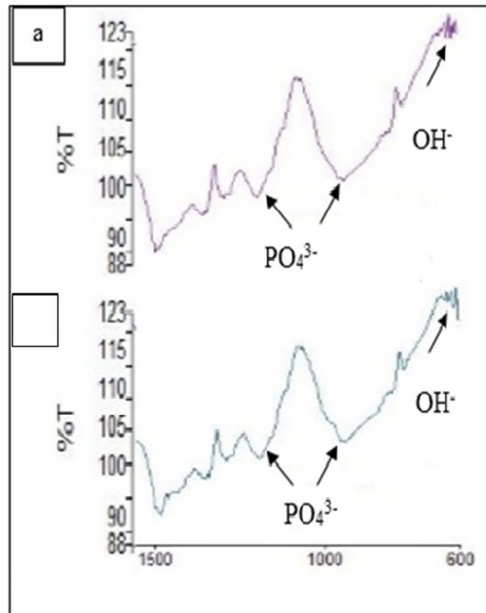


Figure 6: Fourier transform infrared spectra for the coating at different applied voltage

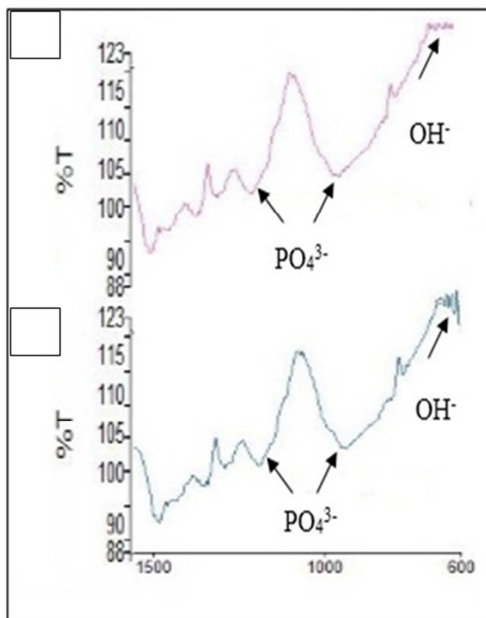


Figure 7: FTIR result for HA coating with deposition time (a) 2 min (b) 17 min

Sample with different deposition time has similar result with increased using voltage. In 2 minutes sample, PO₄ groups appear at wavelength 1194 and 934 cm^{-1} , and hydroxyl group OH⁻ appear at 622 cm^{-1} .

3.4 Corrosion Test

Corrosion resistance value can be determined by using a corrosion test weight reduction methods. The test results will be obtained by the value of corrosion rate (mpy). The smaller value of the rate of corrosion of a metal, will have better corrosion resistance. Figure 6 shows 316L SS coated by HA has smaller value than 316L SS without HA. Rate of corrosion value at different voltage are 4,872, 4,263, and 1,827 mpy. Rate of corrosion value with different deposition time are 4,872, 3,654, 3,045 mpy.

Similar with using voltage, rate of corrosion value increased with increased deposition time. Figure 8 shows rate of corrosion value are 4,872, 3,654, 3,045, and 1,827 mpy.

4.0 CONCLUSION

316L SS coated by HA with higher voltage has better result than lowest voltage. Thickness of HA increased with increased using voltage and has similar result with deposition time. HA coating with variation 80 volt and deposition time 17 minutes has best result for bone plate.

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