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EMPIRICAL MODEL TO PREDICT THE HYDROXYAPATITE THICKNESS ON THE SURFACE OF 316L STAINLESS STEEL BY THE DIP COATING METHOD

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The surface of 316L stainless steel can be coated with hydroxyapatite to overcome its disadvantages as a medical implant, such as low biocompatibility and the release of toxic metal ions in the body after implantation. The coating thickness is one of the important parameters in the coating process. The use of a model to predict the coating thickness is needed to achieve a standard thickness of the coating. This research aim is to determine the empirical model of the hydroxyapatite coating thickness on the surface of 316L stainless steel by the dip coating method. The HA slurry was made by mixing HA powder, polyethylene glycol, and distilled watere using a magnetic stirrer. The 316L stainless steel substrate were dipped into the slurry and then followed by a drying and sintering process. The empirical model of the HA coating thickness on the surface of the 316L stainless steel in this research is y = 406 - 29.5 A - 16 B - 0.588 C + 1.6 AB + 0.0359 AC + 0.0328 BC - 0.00185 ABC with an R2 value of 98.17 %. The empirical model which consists of only significant parameters is y = -101.3 + 1.861 A + 11.774 B. The significant parameters which affect the coating thickness are the amount of HA and the immersion time. The longer the immersion time and the higher amount of HA used, the thicker the coating will be achieved.

INTRODUCTION

The high number of cases of bone injuries have resulted in the increased demand for prostheses, which are artificial components that resemble bone or bone implants. The use of medical implants has grown rapidly over the past few decades because they can increase the life expectancy and improve the implant technologies themselves [1]. Implanted materials can be in the form of metals, ceramics and polymers. Metal has been used as an internal fixation tool to help the healing process of fractured bone tissue. Metals commonly used as implants today include stainless steel, titanium, and cobalt-chromium alloys (Co-Cr alloys) [2,3]. Stainless steel is an alloy steel containing at least 10.5 % Cr. A special characteristic of stainless steel is the formation of a chromium oxide (Cr_2O_3) film layer. This layer characterisation is strong, not easily broken and not visible in plain view. The choice of stainless steel is based on its material properties including its corrosion resistance [4]. The use of 316L stainless steel as a connector or bone substitute is still not perfect because this material still does not have a high biocompatibility with the human body so that its use is still a short-term solution [5]. One of the ways to increase the biocompatibility of 316L stainless steel is by coating it with substances that have high biocompatibility, such as hydroxyapatite.

Hydroxyapatite (HA), which has the molecular formula $Ca_{10}(PO_4)_6(OH)_2$, is a mineral form of calcium apatite that is formed naturally and has the same chemical composition as the original bone tissue [5]. Hydroxyapatite has been recognised as a bone and tooth replacement material because it has a biological similarity to human hard bone tissue [6]. Hydroxyapatite can be synthesised directly from biological material by a calcination process, this is seen from the $CaCO_3$ content which is possessed by high biogenic material, such as chicken eggshells, shells of the mollusc group of animals, shellfish, and animal bones. This synthesis can be carried out by a variety of methods deemed appropriate to the characteristics of the material used [7]. Coating on metal implants with hydroxyapatite has been carried out with various techniques, such as plasma spraying, electrophoretic deposition, sol-gel coating, and dip coating [8].

The dip coating process can be divided into five stages. The first is immersion, where the substrate is put into a coating material solution at a constant speed. The second is the start-up, where the substrate is left in the solution for a few moments and then is removed. The third is deposition, where a thin layer attaches to the substrate by itself when the substrate is removed. Withdrawals are undertaken at a constant speed, which determines the thickness of the coating. The fourth is drainage,

where excess liquid comes out of the surface. The fifth is evaporation, where solvents are evaporated from the solution and produce a thin layer. For volatile solvents, such as alcohol, evaporation occurs during the stages of deposition and drainage [9]. The thickness of the coating can be adjusted by adjusting the concentration of the suspension, the amount of dipping and the variation of the withdrawal speed. Heat treatment of the coated substrate is needed to produce a dense coating layer, strengthen the coating bond and reduce the porosity. However, sintering temperatures that are too high can cause metal substrate degradation and cause hydroxyapatite transformation to a non-crystalline phase which increases the likelihood of a coating release in the bodily fluids [10].

Empirical models are equations obtained from data that can express the relationship between the response and important design factors [11]. Factorial designs are often used in experiments involving several factors where it is necessary to study the combined effects of the factors on the responses. Some special cases of a general factorial design are very important because they are widely used in research, one of which is a case where there are k factors each consisting of two levels called a 2^k factorial design [12]. A 2^3 factorial design is a design for experiments involving three factors, A, B, and C with two levels for each factor. In this design, there are eight treatment combinations. The standard order of treatment combinations in carrying out experiments for these factorial designs are (1), a, b, ab, c, ac, bc, and abc according to the sequence based on Frank Yates. Treatment (1) is when all the factors are at a low level. Treatments a, b, and c are performed to determine the effect of each of the main factors of A, B, and C on the response. The ab, ac, bc, and abc treatments are performed to determine the effect of the two-way and three-way interactions between the factors [11]. The use of computer software is often used in solving statistical methods of a problem. A numbers of software programs are available that can be used to design and analyse experiments, some of which are Design-Expert, Minitab, and JMP. The Minitab and JMP software programs are widely used as statistical software packages [11].

In our previous study, hydroxyapatite was successfully coated onto the surface of 316L stainless steel using the dip coating method by varying the stirring time and the starch mass [13]. The coating thickness is an important parameter in the hydroxyapatite coating on a metal surface. However, the coating thicknesses achieved were still either below the standard or slightly exceeded the standard. Therefore, a model to predict the coating thickness is needed to achieve a standard coating thickness. In this study, the relationship between the slurry composition, immersion time, and sintering temperature to the coating thickness were used to determine the empirical model to predict the hydroxyapatite coating thickness on a 316L stainless steel surface using the dip coating method.

EXPERIMENTAL

Material and Methods

The materials used in this study were hydroxyapatite with a purity of 99.4 % (Lianyungan Kede Chemical Industry co. Ltd, China), 316L stainless steel (Jindal Stainless, India) with dimensions of 2 cm × 3 cm × 0.1 cm, polyethylene glycol (Sigma Aldrich, America), acetone (Merck, Germany), and distilled water (Brataco Chemical, Indonesia).

The HA slurry was prepared by mixing the HA powder with variations of either 18 grams or 24 grams with 54 ml of distilled water and 3 grams of PEG and stirred using a magnetic stirrer with a stirring speed of 300 rpm for 20 hours to produce the slurry. The 316L stainless steel was ground using 1200 grit abrasive paper (Nihon Kensgi Co., Ltd). The stainless steel was then sterilised by immersing it in acetone for 15 minutes, then rinsed with distilled water, and dried using an oven at 110 °C for 10 minutes.

The prepared substrate was dipped once into the HA slurry using the dip coating device with variations of the immersion time of either 10 s or 20 s. The coated substrate was then dried using an oven at a temperature of 110 °C for 30 minutes and sintered for 1 hour with temperature variations of either 700 °C or 800 °C using a furnace (Fugar Mandiri Furnace Type PM 500i). The hydroxyapatite coatings were characterised by Scanning electron microscopy (SEM) (Hitachi Scanning Electron Microscope Type S-3400N) and X-ray diffraction (XRD) (PANalytical X'Pert Pro) analysis. The experiment used a 23 factorial design to evaluate the effects of the immersion time, HA amount and sintering temperature to the HA coating thickness. The achieved HA coating thickness was then analysed using Minitab 19 Software to produce the empirical model.

RESULTS AND DISCUSSION

The dip-coating process, which can be seen in Figure 1, is an important step that affects the quality of the resulting coating. The structure of the film deposited by the dip-coating method depends on the parameters related to the physical properties of the suspension, such as the viscosity and evaporation rate. Generally, a longer immersion time increases the film thickness due to the excellent bond between the molecular monomers of the solution and the substrate. When the substrate has been removed, the solution deposited on the substrate undergoes a rapid and spontaneous change, as the suspension quickly concentrates on the substrate due to the draining force and evaporation of the solvent.

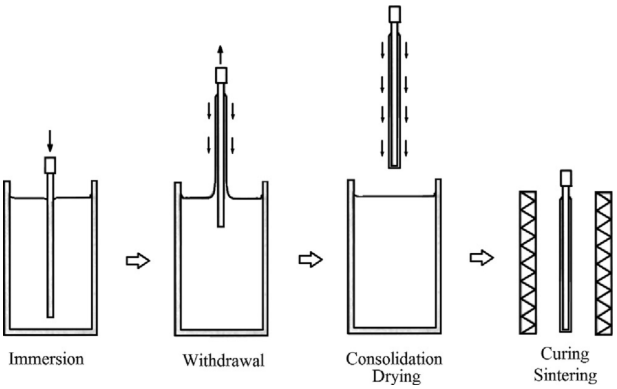

Figure 1. The dip-coating process steps.

SEM Analysis

A SEM analysis was carried out to see the thickness of the hydroxyapatite layer on the surface of the 316L stainless steel substrate at a magnification of 200 ×. In this study, the effect of the variations in the amount of the HA, immersion time, and sintering temperature were studied on the thickness of the HA layer produced on the surface of 316L stainless steel. The results of the SEM analysis of the samples on the various combinations of the HA amount in the slurry, immersion time, and sintering temperature can be seen in Figure 2.

Figure 2a shows the HA layer formed with a thickness of 126 μm when using a slurry with 18 grams of HA, a 10 second immersion time, and a sintering temperature at 700 °C. This thickness increased to 223 μm in Figure 2e when using a slurry with 24 grams of HA at the same immersion time and sintering temperature.

In Figure 2b, a layer with a 130 μm thickness was obtained when the HA amount in the slurry was 18 grams and increased in Figure 2f to 230 μm when the HA amount was 24 grams at the same immersion time and sintering temperature, which were 10 seconds and 800 °C. The same thing happened when the immersion time was 20 seconds and the sintering temperature was 700 °C, that is, in Figure 2c, the layer thickness is 144 μm when the amount of HA in the slurry was 18 grams and increased, as can be seen in Figure 2g, to 242 μm when using 24 grams of HA.

In Figure 2d, an HA layer of 154 μm thickness was obtained when the amount of HA in the slurry was 18 grams, the immersion time was 20 seconds and the sintering temperature was 800 °C. The thickness of the layer increases to 249 μm, as can be seen in Figure 2h, when the amount of HA in the slurry is 24 grams with the same immersion time and sintering temperature.

Data on the increasing hydroxyapatite coating thickness that formed on the 316L stainless steel substrate due to the differences in the amount of hydroxyapatite can be seen in Figure 3.

Based on Figure 3, it can be seen that the larger amount of HA in the slurry, the thicker the HA layer is produced on the surface of the 316L stainless steel substrate. The increase in the coating thickness is due to the thickness of the HA slurry. The greater the amount of HA used, the thicker the HA slurry is produced so that a thicker layer is formed on the surface of the substrate. This is consistent with the previous research that the increase in the layer thickness is affected by an increase in the thickness of the suspension used [10].

The effect of the HA concentration on the thickness of the coating has also been demonstrated in a previous study where a hydroxyapatite coating on the surface of a cobalt alloy using the dip coating method was carried out at three different HA suspension concentrations, namely 0.02 M; 0.04 M; and 0.06 M [14]. The result shows that the higher the concentration of the HA suspension used, the thicker the HA layer is produced.

The thicknesses of the HA layers obtained in this study meet the requirements of the thicknesses of the hydroxyapatite layers, which is 50 - 200 μm [15]. Samples that meet this thickness requirement are samples with the thickness of 126 μm, 130 μm, 144 μm, and 154 μm. Figure 3 shows the SEM result of the morphology of the hydroxyapatite layer on the surface of the 316L stainless steel. Based on the figure, it can be seen that the HA layer covers the surface of the 316L stainless steel substrate. The layer consists of both small and large particles. The amount of HA in the slurry does not affect the morphology of the resulting layer. This is consistent with a previous study that showed HA variations do not produce significant differences on the surface of the HA layer [14].

Figure 4 shows that the surface state of the deposited layer on a variety of HA set ups (a) 18 grams, 10 seconds, 700 °C; (b) 18 grams, 10 seconds, 800 °C; (c) 18 grams, 20 seconds, 700 °C; (d) 18 grams, 20 seconds, 800 °C; (e) 24 grams, 10 seconds, 700 °C; (f) 24 grams, 10 seconds, 800 °C; (g) 24 grams, 20 seconds, 700 °C; (h) 24 grams, 20 seconds, 800 °C, where it can be seen that there are not any significant cavities on the surface of the layer. The cavities are more visible in the lower amounts of HA in Figure 4a, b, c and d. The cavities on the surface of the

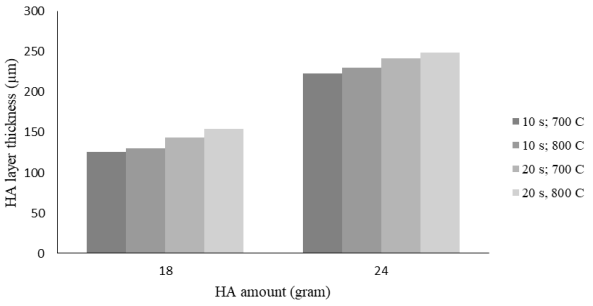

Figure 3. Effect of the hydroxyapatite amount in the slurry on the thickness of the hydroxyapatite layer.

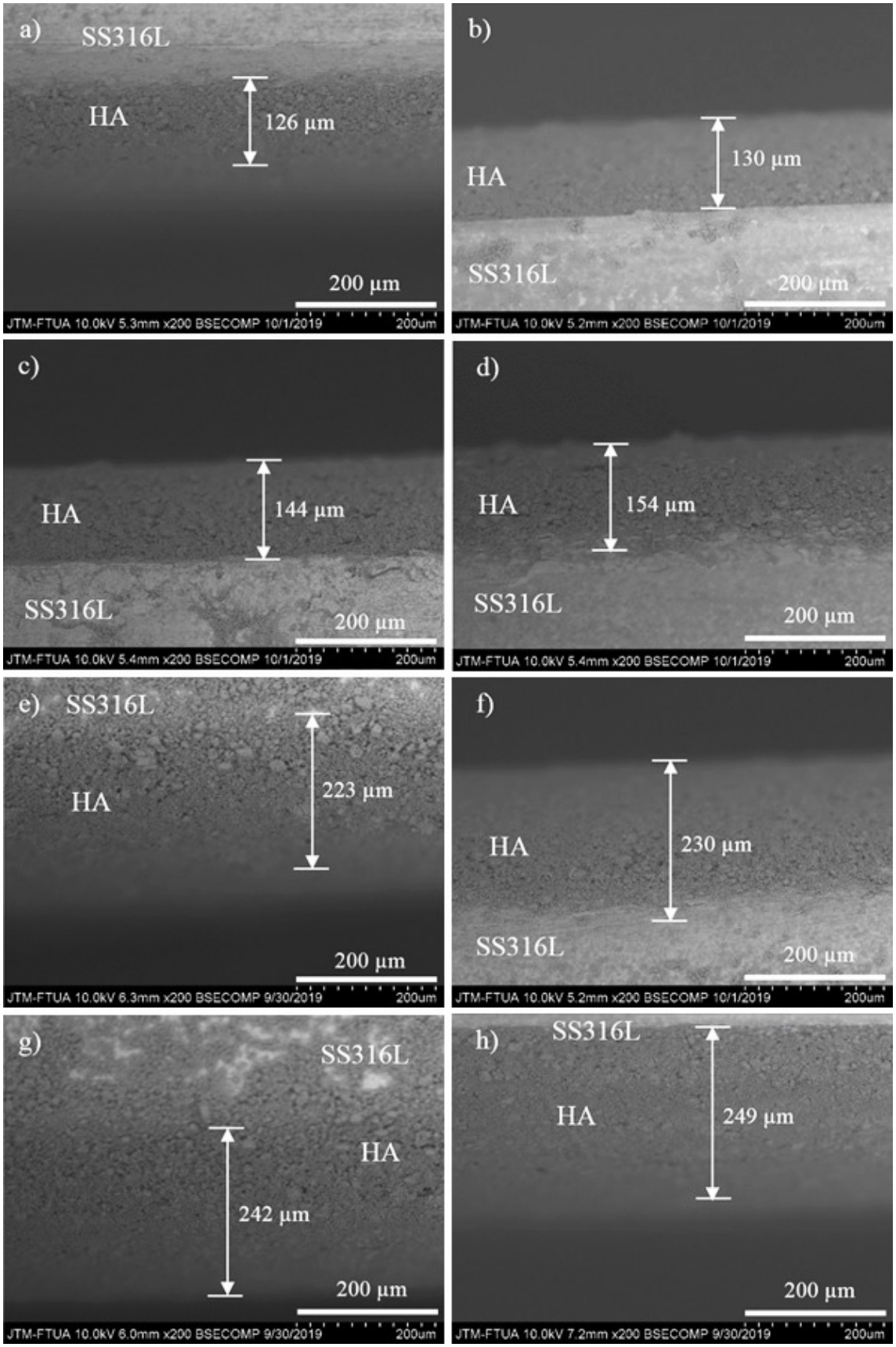

Figure 2. SEM analysis results of the sample layer thickness with variations in the HA amount in the slurry, immersion time, and sintering temperature, respectively: (a) 18 grams, 10 seconds, 700 °C; (b) 18 grams, 10 seconds, 800 °C; (c) 18 grams, 20 seconds, 700 °C; (d) 18 grams, 20 seconds, 800 °C; (e) 24 grams, 10 seconds, 700 °C; (f) 24 grams, 10 seconds, 800 °C; (g) 24 grams, 20 seconds, 700 °C; (h) 24 grams, 20 seconds, 800 °C

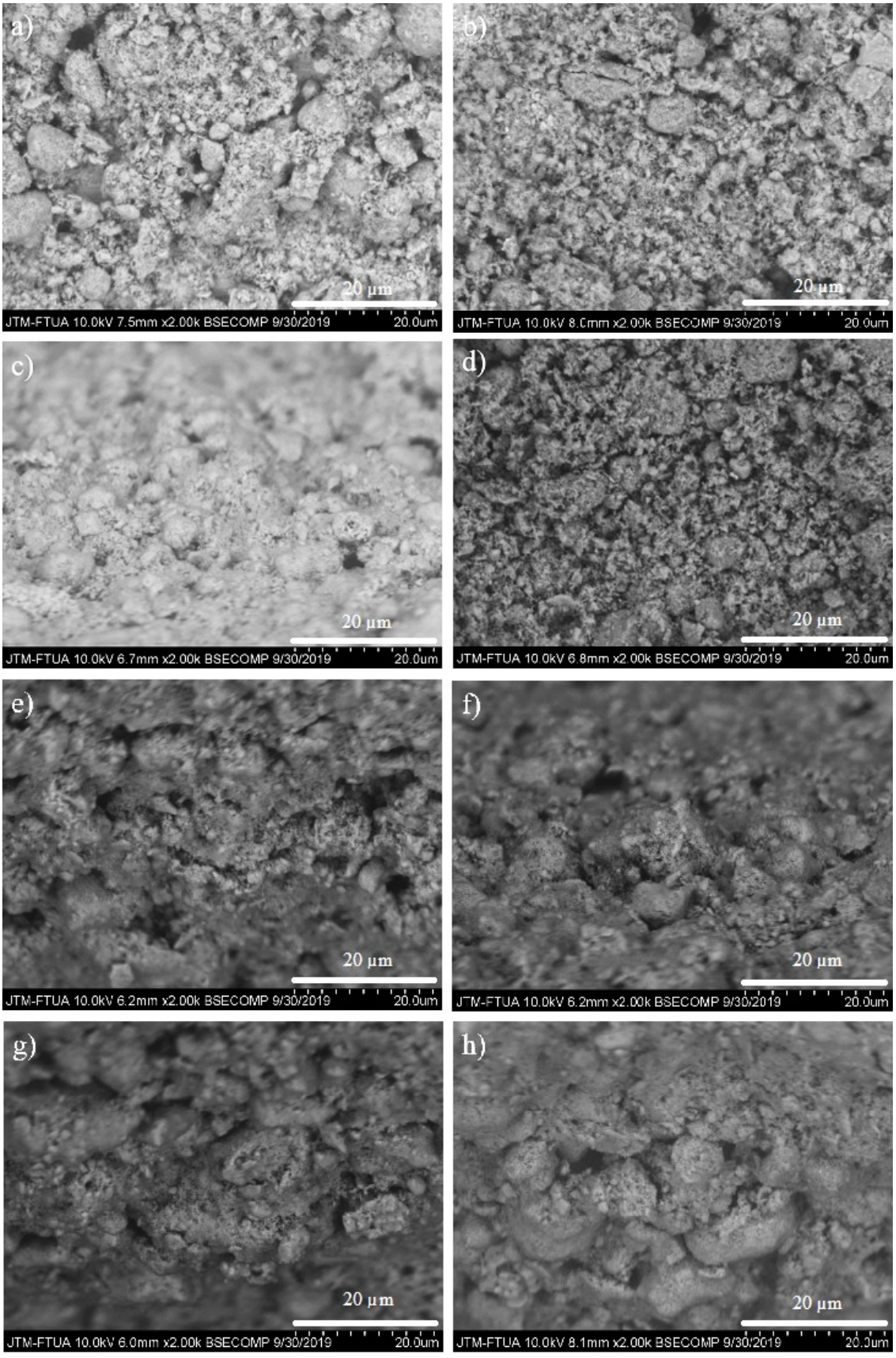

Figure 4. SEM analysis results of the sample layer thickness with variations in the HA amount in the slurry, immersion time, and sintering temperature, respectively: (a) 18 grams, 10 seconds, 700 °C; (b) 18 grams, 10 seconds, 800 °C; (c) 18 grams, 20 seconds, 700 °C; (d) 18 grams, 20 seconds, 800 °C; (e) 24 grams, 10 seconds, 700 °C; (f) 24 grams, 10 seconds, 800 °C; (g) 24 grams, 20 seconds, 700 °C; (h) 24 grams, 20 seconds, 800 °C

coating are the result of a burning Polyethylene Glycol (PEG) on the surface. It can be seen that a higher amount of hydroxyapatite coats the entire surface of the substrate in the absence of cracking.

The immersion time was varied between 10 seconds and 20 seconds, which is based on our previous study [13]. The data of the HA layer thickness formed on the 316L stainless steel substrate affected by the immersion time at each slurry composition and sintering temperature can be seen in Figure 5.

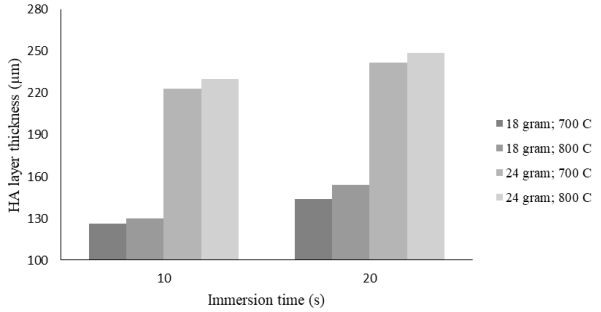

Figure 5. Effect of the immersion time on the hydroxyapatite coating thickness.

Based on Figure 5, it can be seen that the immersion time affects the thickness of the HA layer. The longer the immersion time of the 316L stainless steel substrate in the slurry, the thicker the resulting HA layer is. The thickness of the layer increases with an increasing immersion time [16]. The longer the immersion time, the longer the contact that occurs between the 316L stainless steel substrate with the HA slurry, so that the resulting layer is thicker.

In this study, the sintering temperature was varied between 700 °C and 800 °C following our previous study [13]. The data of the hydroxyapatite layer thickness formed on the 316L stainless steel substrate affected by the sintering temperature can be seen in Figure 5.

Based on Figure 6, it can be seen that the sintering temperature affects the thickness of the hydroxyapatite layer on the surface of the 316L stainless steel. The higher the sintering temperature, a thicker HA layer is produced. The thickness difference between the two temperatures is small and insignificant when compared to the effect of the amount of HA and the immersion time of the substrate. This is in accordance with a previous study that showed that the effect of the sintering temperature on the HA layer thickness is not significant [17].

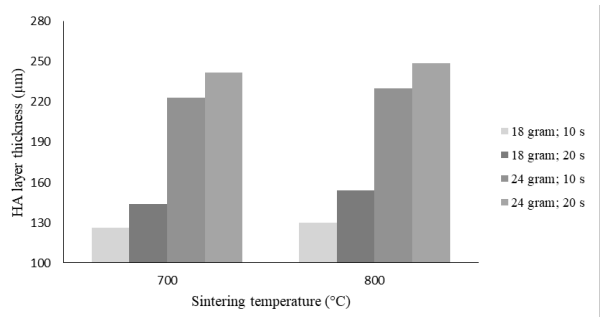

Figure 6. Effect of the sintering temperature on the hydroxyapatite coating thickness.

Empirical model of the HA coating thickness

The coating thickness is one of the important parameters in the coating of bioactive materials on the surface of metal implants. This study uses 2 levels and 3 factors – a (23) factorial design - to determine the coating thickness model on an HA coating on 316L stainless steel surfaces and evaluate the effect of the dipping time (A), the amount of HA in the slurry (B), and the sintering temperature (C) on the HA coating thickness. The factors and levels in this study can be seen in Table 1.

Table 1. Factors and levels of the 2^3 factorial design.

Factor	Unit	Level (Uncoded)		Level (Coded)	
		Low	High	Low	High
Immersion time (A)	S	10	20	-1	+1
HA amount (B)	Gram	18	24	-1	+1
Sintering temperature (C)	°C	700	800	-1	+1

The factors and levels in Table 1 were chosen so that the thickness of the obtained layer can meet the requirements of the hydroxyapatite layer, which is 50 - 200 µm (Heimann, 2002). Each treatment was carried out with two replications. Data on the thickness of the HA coating can be seen in Table 2.

Table 2. Design matrix and HA coating thickness results.

Treatment	Factor			Coating Thickness (µm)	
	A	B	C	Replication 1	Replication 2
(1)	-1	-1	-1	128.25	133.62
a	+1	-1	-1	140.23	143.48
b	-1	+1	-1	203.27	178.54
ab	+1	+1	-1	219.64	220.61
c	-1	-1	+1	132.86	134.80
ac	+1	-1	+1	149.66	145.08
bc	-1	+1	+1	207.90	196.89
abc	+1	+1	+1	221.42	224.87

The model of the HA coating thickness on the 316L stainless steel substrate is obtained by processing the research data using the statistical software Minitab 19. The results of the statistical analysis using Minitab 19 can be seen in Table 3.

Table 3 is the result of the statistical analysis of the research data that shows the estimated effects, coefficients of the regression model, and p-values of each parameter and a combination of parameters. The coefficients are quantitative measures of the effect of the parameters. The higher the coefficient value, the greater the effect of these parameters on the thickness of the HA layer [16].

Table 3. Results of the statistical analysis using Minitab 19.

Term	Effect	Coef	P-Value
Constant		173.82	0.000*
A (Immersion time)	18.61	9.30	0.001*
B (HA amount)	70.65	35.32	0.000*
C (Sintering temperature)	5.73	2.87	0.146
A*B	6.38	3.19	0.111
A*C	-1.46	-0.73	0.692
B*C	1.52	0.76	0.679
A*B*C	-2.77	-1.39	0.458

R^2 = 98.17 %; Adj. R^2 = 96.56 %;
* significant at a significance level of less than 0.05

The p-value represents the level of significance of the parameter. Parameters with p-values less than 0.05 indicate that the parameters have a significant effect on the model produced with a significance level of less than 0.05. Based on Table 3, the parameters that have a significant influence on the thickness of the HA layer on the 316L stainless steel substrate in this study are the dipping time (A) and the amount of HA (B). While the sintering temperature (C) and the two-way and three-way interactions between the parameters have no significant effect on the thickness of the HA layer.

The coefficient of determination (R^2), adjusted coefficient of determination (Adj. R^2), and p-value are used to evaluate how well the fit and adequacy of the model is. Based on the analysis, the R^2 value of the model obtained was 98.17 %. This value shows a very good match, so that a 98.17 % variation in the coating thickness can be predicted using this model.

Figure 7 shows a pareto chart that states the significance of the parameters to the thickness of the layer. Based on the figure, the limit on the standardisation of the effects on this model is 2.31. Parameters with an effect standardisation value more than 2.31 are parameters that significantly affect the thickness of the HA coating. While parameters with effect standardisation values less than 2.31 do not significantly affect the coating thickness. Based on the chart, it is known that the parameters that most significantly affect the thickness of the HA layer are the amount of HA in the slurry and the immersion time.

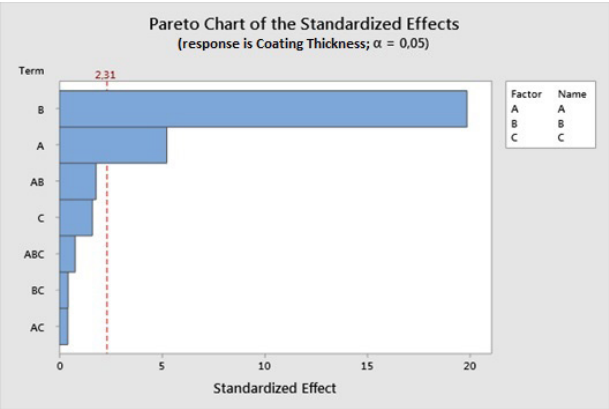

Figure 7. Pareto chart of the standardised effects.

While the sintering temperature and interaction between the parameters did not significantly affect the thickness of the HA layer.

An empirical model of the HA coating thickness on the 316L stainless steel substrates based on the regression model from the results of the statistical analysis can be seen in Equation 1.

$$y = 406 - 29.5\,A - 16\,B - 0.588\,C + 1.6\,AB + \\ + 0.0359AC + 0.0328\,BC - 0.00185\,ABC \quad (1)$$

Equation 1 shows an empirical model that is composed of all the parameters, namely the main parameters, the two-way interactions, and the three-way interactions. Empirical models that are only composed of the significant parameters can be seen in Equation 2.

$$y = -101.3 + 1.861\,A + 11.774\,B \quad (2)$$

The predicted value of the HA coating thickness on the surface of the 316L stainless steel can be calculated using an empirical model consisting of all the parameters so that the match is higher. The coating thickness values based on the experiment and prediction obtained using the model can be seen in Table 4.

Table 4. Experiment and predicted value of the coating thickness.

Immersion Time (s)	HA Amount (gram)	Sintering Temperature (°C)	Coating Thickness (μm)	
			Experiment	Model
10	18	700	128.25	130.88
20	18	700	140.23	142.08
10	24	700	203.27	190.94
20	24	700	219.64	220.44
10	18	800	132.86	133.72
20	18	800	149.66	147.52
10	24	800	207.90	202.36
20	24	800	221.42	223.36
10	18	700	133.62	130.88
20	18	700	143.48	142.08
10	24	700	178.54	190.94
20	24	700	220.61	220.44
10	18	800	134.80	133.72
20	18	800	145.08	147.52
10	24	800	196.89	202.36
20	24	800	224.87	223.36

Based on Table 4, it can be seen that the value of the coating thickness between the results of the experiment and the model has small differences. The normal probability plot as a residual analysis can be seen in Figure 8.

Based on Figure 8, it can be seen that the data is normally distributed, it spreads following a diagonal line pattern. Based on the coefficient of determination (R^2) and the residual analysis, it shows that the model

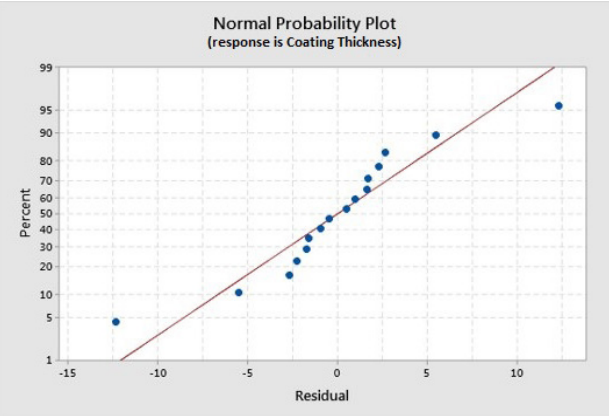

Figure 8. Residual analysis of the normal probability plot.

obtained can be used to predict the thickness of the HA coating at the boundary conditions within the research parameters.

XRD analysis

An XRD analysis was carried out to determine the crystalline phase of the layer formed on the surface of 316L stainless steel. Figure 8 shows the XRD diffractogram of the HA layer on the 316L stainless steel surface. Based on data from the International Center for Diffraction Data (ICDD), the characteristic value of the standard XRD HA analysis results with the reference codes 01-072-1243 are (002), (211) and (112) with an angle of 2θ 25.875 °; 31.741 °; and 32.179 °.

The XRD analysis results show that no phases other than HA were formed in the coating on the surface of the 316L stainless steel. This shows that the sintering temperatures used, which were 700 °C and 800 °C, do not cause the decomposition of HA into another tricalcium phosphate phase. This is consistent with a previous study where the HA coating process on titanium surfaces with a sintering temperature of 800 °C showed that only the HA phase was detected in the layer and that the HA was not decomposed [18]. Other tricalcium phosphate phases can occur due to the HA decomposition at high sintering temperatures [19].

Based on Figure 9a and b, it can be seen that the intensity of the diffractogram peaks increases when the sintering temperature increases. An increase in the diffractogram intensity also occurs when the amount of HA in the slurry increases which can be seen in Figures 9a and c. The intensity of the diffraction is related to the crystallinity. The increase in the intensity shows an increase in the crystalline phase of the HA [18]. The higher the sintering temperature, the higher the crystallinity of the HA layer is. This happens because the sintering process was carried out at the HA recrystallisation temperature, which is 502 - 1002 °C [20]. At this temperature, the HA constituent atoms will move and are arranged regularly to form a better crystal

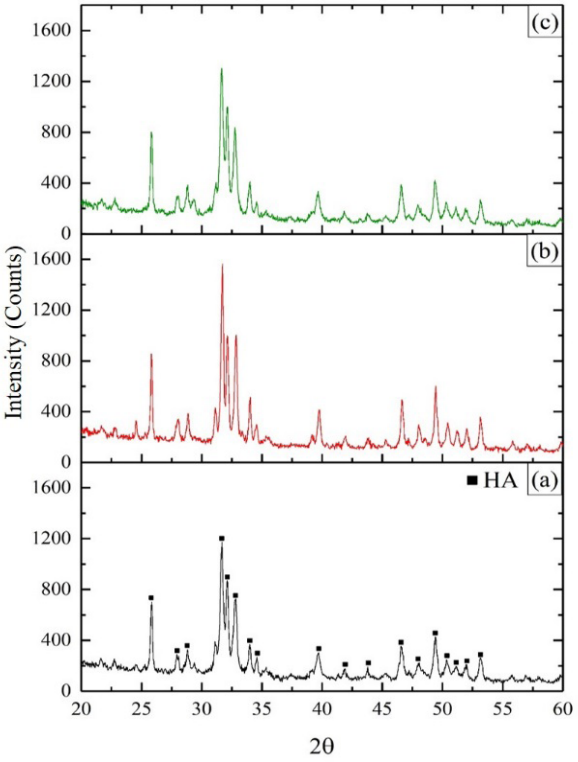

Figure 9. XRD diffractogram of the HA coating on the surface of the substrate with a) 18 grams, 10 seconds, 700 °C; b) 18 grams, 10 seconds, 800 °C; c) 24 grams, 10 seconds, 700 °C.

structure. So, it can be concluded that the higher the sintering temperature, the higher the crystallinity of the formed HA layer is [14].

CONCLUSION

Hydroxyapatite has been successfully coated onto the surface of a 316L stainless steel substrate. The empirical model of the hydroxyapatite layer thickness on the surface of the 316L stainless steel is y = 406 - 29.5 A - 16 B - 0.588 C + 1.6 AB + 0.0359 AC + 0.0328 BC - 0.00185 ABC with an R^2 value of 98.17 %. The empirical model which is only composed of parameters that significantly affect the thickness of HA is y = -101.3 + 1.861 A + 11.774 B. The parameters that significantly affect the thickness of the coating are the amount of HA in the slurry and the immersion time. The longer the immersion time and the higher amount of HA in the slurry, the thicker the resulting layer is.

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