Hydroxyapatite Coating on Stainless Steel 316L using Flame Spray Technique

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Hydroxyapatite Coating on Stainless Steel 316L using Flame Spray Technique

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ABSTRACT

This study was a preliminary study on flame spray coating with hydroxyapatite (HAp). Coating is one of the technique to improve metal resistance to corrosion. In this study, flame spray coating using HAp was performed on stainless steel 316 L as a material for medical devices. This synthetic compound contains elements which are biocompatible and bioactive in human body where they can stick to body tissues or muscles.HAp has been extensively used as a bone substitute because of its crystal structure, biocompatibility and osteoconductive nature. In this study, 316L SSwas coated by HAp using flame spray method with varied oxygen flowrate and air pressure. The result of this study showed that the air pressure of 1 bar and oxygen flowrate of 25 l/min had the thickest coating which was 123.5μm and the lowest corrosion rate which was 0.0261 mm/year. The air pressureof 3 bar and oxygen flowrate of 35 l/min produced the lowest thickness which was 32.5μm and the highest corrosion rate which was 0.0761 mm/year. The use of high air pressure and oxygen flowrate decreased the coating thickness and the corrosion rate. The result revealed that flame spray method was effective to be used to coat HAp on 316L SS.

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1. INTRODUCTION

Biomaterial is used to create surgical implants and devices to replace parts or the function of body organs safely and economically. Metal types that are commonly used are Ti alloys, Co-Cr alloys and stainless steel. The type of stainless steel which is generally used for implants is 316L SS. The 316L SS needs to be protected to restrain corrosion rate because even though it is a biomaterial implant; it still corrodes in the human body. ASTM has standardized stainless steel for implants in standard ASTM F13 [1], F899 [2] and F2181 [3]. The metal has been selected because of its structural function and inertness (having no ability to react with environment). However, in the newest development, implants are expected to possess bioactivity and bio functionality such as being compatible with blood (hemocompatible) and surface modifiable. Therefore, the metal is coated with HAp to make it conducive with human bones [4]. HAp is mostly applied in medical field especially for bone implants; because, it has very similar characteristics with components of human bones and teeth. Besides being applied as material for bone implants, HAp is also used as coating material for metal which is implanted into human body. HAp is biocompatible and bioactive with human body where it can stick to body tissues or muscles. The coating process is performed by depositing HAp on the substrate surface. Previous studies revealed that HAp is effective in increasing corrosion rate of 316L SS using some methods applying functionalized multiwalled carbon nanotubes (f-MWCNT)[5], nano particles [6], tissue engineering [7], and low velocity oxyfuel (LVOF) spray [8]. One of the coating methods which can be done is flame spray coating. Flame spray coating is one of the coating techniques which can use the most varied materials and geometries to coat [9]. Flame spray coating can also be defined as sticking coating material using a special

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instrument that is able to melt and push the material onto the substrate surface which has been prepared previously. One of the types of flame spray coating is powder flame spray where the heat is produced from fuel combustion with oxygen. In this case, the fuel being used is acetylene. The feedstock being used is in the form of powder [10]. Some previous studies were conducted to improve the quality of metal surface and one of the approaches is to perform coating. On CrCr/NiCr coating with the method of HVOF spraying, the setting of powder feed rate, stand-off distance and gun barrel length can affect the temperature of the particles. Stand off distance and oxygen flow rate are the main factors which affect the molten particle speed [11]. Determining the level of pressure in coating process with high velocity air fuel method (HVAF) can affect particle speed of the particle, flame temperature and coating thickness (deposition efficiency [12]. The factor of coating thickness can effect on corrosion resistance where thicker coating makes better protection from corrosion [13]. The purpose of the study is to know the effect of pressure and oxygen flow rate in conventional flame spray coating on coating thickness and corrosion rate. The findings of this study is the right parameter in the use of flame spray in HAp coating. We expect that this study can be reference in the continuation of the use of flame spray in biomaterial coating.

2. MATERIALS AND METHODS

2. 1. Materials The specimen in the study was 316L SS. Furthermore, its compositions is written in Table 1.

The roughness of the specimen surface without treatment had Ra value of 5.482μm. The specimen dimension is displayed in Figure 1.

TABLE 1. Compositions of 316L SS (% weight)

Composition	Weight Percentage
C	0.023
Mn	1.31
Si	0.34
P	0.032
S	0.001
Cr	16.7
Mo	2.02
Ni	10.1
N	0.04
Fe	69.434

Figure 1. The specimen dimension

The formula of HAp that was used in the study was $HCa_5O_{13}P_3$. It had melting point of 1100°C, density of 3.140 g/cm^3 and the particle size of 30 µm.

2. 2. Flame Spray Coating Flame spray coating was performed by using Metco 5P-II combustion powder spray gun with varied oxygen flow rate: 25, 30 and 35 l/min and varied air pressure: 1, 2 and 3 bars. Substrate surface s peed during the coating process was made constant with the fuel flow rate staying at 25 l/min. The flame spray tool was equipped with Matco 5P-II combustion powder spray gun. The process of gas consumption used acetylene ranging from 13.5 – 31.5 NLPM, hydrogen ranging from 90 – 144 NLPM, oxygen ranging from 20 – 45 NLPM and compressed air of 0.85 m^3/min at 4.5 bars. Air requirements used was the standard of air cap which was 10 to 15 psi, with pinch air cap of 5 psi in maximum, cooling air of10 – 20 psi, and air jet assembly of 50 – 90 psi. Flame temperature of the gun produced 2500- 3750^{o}C.The spray distance was 120 mm at an angle of 90°. The coating was performed to make two coats. The result of experiment can be seen in Figure 2.

2. 3. The Coating Thickness Measurement The coating thickness was measured using NOVOTEST TP-1M. Novotest TP-1M is able to measure the thickness of the coating on ferrous and non ferrous metal. Probe used to measure the coating thickness on Ferous metal was F 0.5 max 500 ɥm).Probe used to measure the coating thickness on non Ferous metal was max 2000 μm. After the coating process, coating thickness was measured. The

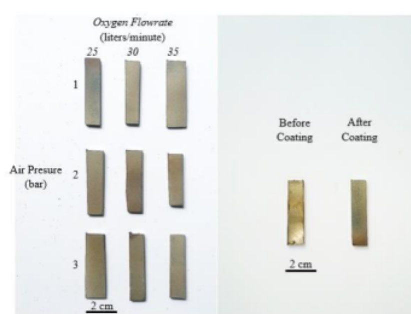

Figure 2. Specimen after coating

measurement was performed in these sequences. First, it was done by pressing the on button on the coating thickness gauge. Then, the probe stylus was directed to the surface of the work piece. Next, the stylus was pressed gently on the surface of the specimen until the display showed the measurement result. The last step was to make note about the measurement result of each coated specimen.

2. 4. The Potentiodynamic Polarization Test The measurement of corrosion rate used Hank's balanced salt solution (HBSS) with pH specification 7.4 ± 0.2, osmolality 290 ± 20 mOsm and Endotoxin < 0.1 EU/ml. This is an artificial blood condition.

The potentiodynamic polarization measurement was performed at room temperature (298K).The Autolab PGSTAT 204N functioning as a potential source was set at ± 0.1 mV and the scan rate was 0.001V/s. Three-cell electrode was set with the workpiece as the working electrode, platinum as the counter electrode and Ag/AgCl (KCl 3 M) as the reference electrode. The coated specimen was exposed into HBSS solution for 1 cm^2. Autolab PGSTAT 204 N was connected with software NOVA 11.0. In the measurement, workpiece density, molecular weight and surface width were added. In order to calculate the parameters of the corrosion such as Icorr, βa and βC, the polarization curves were extrapolated.

Corrosion rate or a rate at which a material is taken as a consequence of a chemical action, which is an important parameter stated as corrosion penetration rate (corrosion penetration rate/CPR) or the thickness of a material which is lost per unit time for the given equation:

$$\text{CPR} = \frac{KW}{\rho At} \quad (1)$$

where,W is the lost weight after contact over time (t), ρ is density of material, A is the width of a specimen which is exposed and K is a constant which the amount depends on the unit system used.

Based on Faraday's law, the corrosion rate was obtained in Equation 2.

$$CR(mpy) = \frac{0.129 \times I_{corr} \times EW}{\rho} \quad (2)$$

where, I_{corr} is current density, EW is equivalent weight. I_{corr} was calculated by using Equation 3.

$$I_{corr} = \frac{b_a \times b_c}{2.3(b_a + b_c)} \times \frac{1}{R_p} \quad (3)$$

2. 5. The Surface Roughness Measurement Surface roughness measurement was performed using Mitutoyo Surface Roughness Tester SJ-210.

2. 6. The Morphology of the Surface Micrograph of the specimens with coating was measured using Scanning Electron Microscope (SEM). The type of SEM used was Phenom G2 Pro. The SEM measurement was performed in cross section.

3. RESULTS AND DISCUSSIONS

The data of HAp coating thickness measurement of 316L SS with variations in pressure and oxygen flowrates are displayed in Figure 3.

Figure 3 displays the data of coating thickness measurement with variations in pressure. It is shown that increasing the pressure can decrease the coating thickness.

This happens because high pressure will increase the particle speed of HAp particles which reduces time to heat the particles [12]. This leads to a condition where HAp particles are not heated well and cannot melt perfectly. The result is that the particles do not stick to the substrate surface maximally [14].

3. 1. The Effect of Oxygen Flowrate on Coating Thickness The measurement of coating thickness produced from varied oxygen flowrate shows that increasing the oxygen flowrate can decrease the coating thickness, as shown in Figure 4.

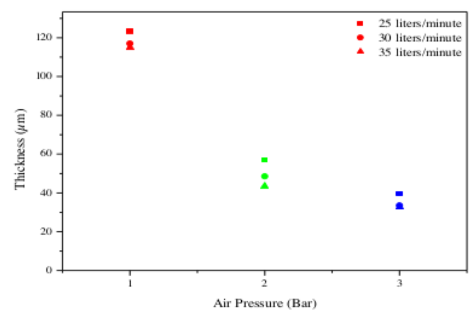

Figure 3. The effect of pressure on coating thickness

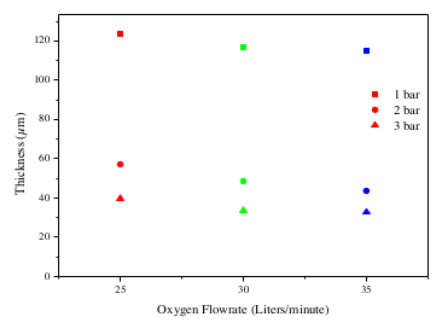

Figure 4. The effect of oxygen flow rate on coating thickness

The thickest coating was obtained at 25 l/min of fuel flow rate. The imbalanced amount of fuel flow rate and oxygen flow rate decreases the flame temperature of the torch. This causes the heating of HAp particle not maximum and thus the particles do not melt and stick to the substrate surface [15]. An increase in oxygen flow rate causes a decrease in flame temperature and it produces higher porosity level and the abundant particles melting which causes lower bound strength. The use of too high oxygen flowrate decreases the coating thickness.

The increase of oxygen flowrate will increase the flame temperature which can melt the HAp particles. However, the abundant use of oxygen flowrate can decrease the flame temperature. Consequently, the HAp particles do not melt perfectly and cannot stick to the substrate surface [11, 15].

3. 2. The Effect of Pressure on Surface Roughness

Figure 5 presents the result of the surface roughness measurement with respect to pressure variation. It is seen that surface roughness values are mostly similar at all variations of pressure. The values are not different significantly even though they were produced at different variations of the pressure.

The increase in flame temperature and molten particle speed causes the particles dispersion on the substrate surface and thus produces low roughness value [8]. However, the surface roughness values produced in this study are not significantly different. This is because the composition of the fuel and the oxygen at pressure of 3 bars produces flame with higher temperature. Thus, much HAp melts. The best pressure is not the higher one, but the most optimum. However, higher pressure does not always produce a lower coating roughness. At low pressure, HAp will produce fine particles. High pressure enables much HAp melts which leads to high roughness [16].

Coating roughness which was produced from varied pressure of 1, 2 and 3 bars with 25 l/min of oxygen flowrate has Ra values of 4.553, 4.376 and 4.532 µm, respectively. The use of oxygen flowrate of 30 l/min has surface roughness values of 4.020, 4.688 and 4.629 μm. Then, the use of oxygen flowrate of 35 l/min has values of 4.603, 4.580 and 4.686 µm .The results show that the flame spray coating is not able to produce even coating, especially when it is performed manually by human.

The surface roughness especially on the coating result provides stabilization during the implant placement until the bone can grow and adhere to the implant surface to increase the implant bonding.

3. 3. The Effect of Pressure and Oxygen Flowrate on Corrosion Rate

The tafel plot of different pressures and oxygen flowrates effect on corrosion rate are illustrated in Figures 6-8.

In Figure 6, it can be seen that the lower the pressure is, the more the tafel curve slides down. Tafel curve which slides down indicates the decrease of Icorr value and thus the corrosion rate gets lower [17]. Figures 7 and 8 display the results of potentiodynamic polarization test

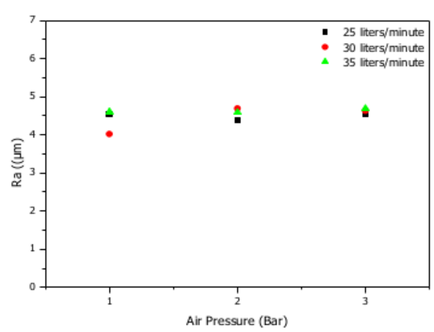

Figure 5. The effect of pressure on surface roughness

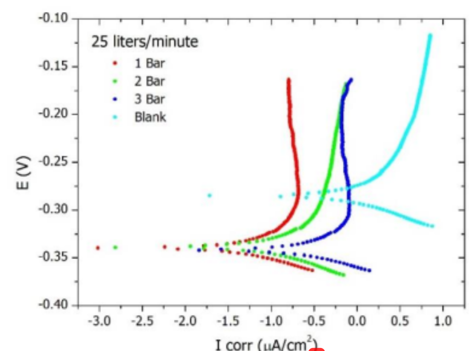

Figure 6. The tafel curve of pressure and oxygen flowrate with variation of 25 l/min

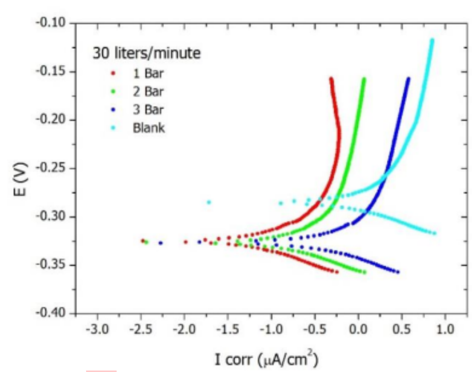

Figure 7. The tafel curve of pressure and oxygen flowrate with variation of 30 l/min

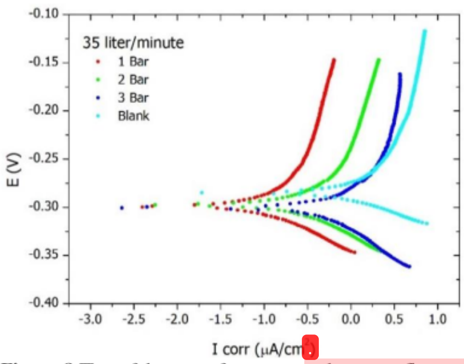

Figure 8. The tafel curve of pressure and oxygen flowrate with variation of 35 l/min

of the specimens which had been treated with flame spray. From curves in Figures 6- 8, it can be compared that the current density is low at overall potential range on all coated materials.This indicates that the corrosion rate on the substrate decreases. Low current density will produce low corrosion rate as well [18, 19]. In the oxygen flow rate variation of 0.25 l/min, the coating results have better passivation ability than other variations. This shift to the right or in the passive direction indicates that there is an increase in electron retention activity which attacks (cuases corrosion) where corrosion activity becomes passive on the coating result compared to that without coating [20]. Corrosion rate depends on the current density which appears. The corrosion rate will be higher if the current density is higher. The parameter values of potentiodynamic polarization of the Tafel curve in Figures 6-8, also can be seen in Table 2.

TABLE 2. The Data of Potentiodynamic Polarization Parametermodel

Pressure (bar)	Oxygen flowrate (liters/minute)	βa (V/dec)	Bc (V/dec)	Ecorr (V)	Icorr (qA/cm²)	CR (mmpy)
Blank		0.0935	-1.19	-0.3249	7.7050	0.08154
1	25	0.0390	0.071	-0.3394	2.4674	0.02611
2	25	0.0416	0.095	-0.3745	2.4888	0.02634
3	25	0.0440	0.149	-0.3412	3.4224	0.03622
1	30	0,0593	0.079	-0.3240	2.6994	0.02857
2	30	0.0620	0.135	-0.3253	3.5456	0.03752
3	30	0.0548	0.194	-0.3781	3.6430	0.03855
1	35	0.0816	-1.13	-0.2991	4.7869	0.05066
2	35	0.1021	0.326	-0.3613	5.0841	0.05380
3	35	0.5916	0.171	-0.3008	7.1888	0.07608

The decrease in corrosion rate is caused by the use of low pressure. If the pressure is getting lower, the particle speed of the HAp part will be slower and thus gives longer time toget to the substrate surface [12]. The effect is that HAp particles can be heated and can stick better to the substrate surface. This will increase the coating thickness which protects the substrate from the environment and decrease the corrosion rate. The measurements of coating thickness and corrosion rate support each other. At low pressure, the particles produced are finer and it leads to smoother surface. The increase in spray distance leads to fine size of the particles on the surface. Bigger particles tend to form big droplets with the volume which partly melts [21]. Closer distance of the gun will produce less smooth or rough surface. This is because the particles have not melted perfectly. Thus, when reaching the substrate surface, the particle size is still round. The roundness can produce hollows on the surface of the coating result which causes higher roughness value and porosity [22]. The variation of oxygen flowrate and air pressure are able to prevent the electrolite ion of HBSS or electrons to get in to the surface of 316L SS.

The decrease in corrosion rate happens because of more balanced use of oxygen flowrate and fuel flowrate. In this study, the fuel flowrate used was 25 l/min. The decrease in the use of oxygen flowrate will increase the flame temperature because the oxygen flowrate and fuel flowrate are more balanced, and thus the heating of HAp particles is better. The effect is that the particles can stick better to the substrate surface [14, 23, 24]. This results in the increase in coating thickness which protects the substrate from the environment and decreases the corrosion rate. This can be seen from the result of micro photograph in Figures 9-10.

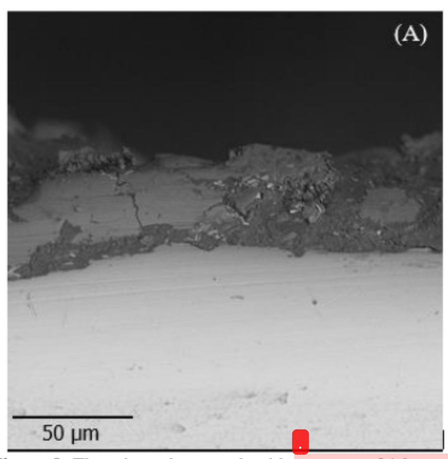

Figure 9. The micro photograph with pressure of 1 bar and oxygen flowrate of 25 l/min

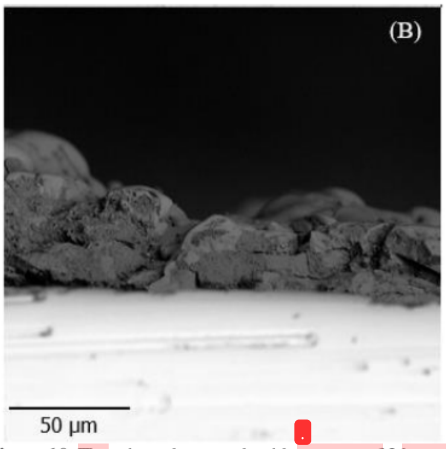

Figure 10. The micro photograph with pressure of 3 bar and oxygen flowrate of 35 l/min

Figure 10 presents the specimen which has been treated with pressure of 1 bar and oxygen flowrate of 25 l/min of oxygen flowrate. In Figure 11, the specimen is given pressure of 3 bars and oxygen flowrate of 35 l/min. From the coating result, it can be known that the specimen in Figure 9 has solid coating while the specimen in Figure 10 has more porous coating which is not as solid as the specimen in Figure 9. In another words, specimen in Figure 10 has better coating quality compared with the specimen in Figure 10 [25]. It is also found that the specimen in Figure 9 has the lowest corrosion rate while the specimen in Figure 10 has the highest corrosion rate. The coating result influences the corrosion rate.

4. CONCLUSION

The result of this study showed that variation of pressure of 1 bar and oxygen flowrate of 25 l/min produced the thickest coating which is 123.5µmand the lowest corrosion rate which is 0.0261mm/year. The variation of pressure of 3 bars and oxygen flowrate of 35 l/min produces the lowest thickness which is 32.5µm and the highest corrosion rate which is 0.0761 mm/year.

The use of high pressure and oxygen flowrate will decrease the coating thickness and the corrosion rate. This is because high pressure increases the particle speed of HAp particles. This makes much HAp metal meltand agglomerate on the surface. The result is that the nonhomogeneous and porous coating. The porosity triggers higher corrosion rate on the coating. This condition will reduce time to heat HAp particles and thus the particles are not heated well and cannot stick to the substrate. This results in the porous coating and the specimen is not protected well from the environment.The specimens which are coated with HAp produces lower corrosion rate compared to those were not reported. This study can initiate future research on flame spray coating on biomaterial.

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Persian Abstract

چکیده

این مطالعه یک مطالعه مقدماتی روی پوشش اسپری شعله با هیدروکسی آپاتیت (HAp)بود. پوشش یکی از تکنیک های بهبود مقاومت فلز در برابر خوردگی است. در این مطالعه ، پوشش اسپری شعله با استفاده از HAp بر روی فولاد ضد زنگ ۳۱۶ L به عنوان ماده ای برای دستگاه های پزشکی انجام شد. این ترکیب مصنوعی حاوی عناصری است که در بدن انسان سازگار و زیست فعال هستند و در آنجا می توانند به بافت های بدن یا عضلات بچسبند. از HAp به دلیل ساختار بلوری ، سازگاری زیستی و ماهیت استخوان سازی به عنوان جانشین استخوان استفاده گسترده ای شده است. در این مطالعه استیل ، ۳۱۶ L با استفاده از روش اسپری شعله با جریان متنوع اکسیژن و فشار هوا توسط HAp پوشانده شد. نتیجه این مطالعه نشان داد که فشار هوا ۱ بار و جریان اکسیژن ۲۵ لیتر در دقیقه دارای ضخیم ترین پوشش که ۱۲۳/۵ میکرومتر بود و کمترین میزان خوردگی آن ۰.۲۶۱ میلی متر در سال بود. فشار هوا از ۳ بار و جریان اکسیژن ۳۵ لیتر در دقیقه کمترین ضخامت را نشان داد که ۳۲/۵ میکرومتر بود و بیشترین میزان خوردگی را ۰/۰۷۷ میلی متر در سال نشان داد. استفاده از فشار هوای بالا و جریان اکسیژن باعث کاهش ضخامت پوشش و میزان خوردگی می شود. نتیجه نشان داد که روش اسپری شعله برای استفاده برای پوشاندن HAp روی استیل 316L موثر بود.

Hydroxyapatite Coating on Stainless Steel 316L using Flame Spray Technique

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