Ghosh and Mitra, J Material Sci Eng 2013, 2:1
DOI: 10.4172/2169-0022.1000118

Journal of Material Sciences & Engineering

Review Article Open Access

Effectiveness of Dicalcium Phosphate Dihydrate as Biocompatible Coatings on 316L and 316LN Stainless Steel

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Abstract

The corrosion behaviors of two materials 316L Stainless Steel and 316LN Stainless Steel have been investigated for use as biomaterials. These samples were electrophoretically coated with Dicalcium phosphate dihydrate, and dip coated with polyvinyl alcohol. Time, current, concentration and voltage were the variables during electrophoresis. Dip coating was done for the same periods of time as was done during electrophoresis. Corrosion resistance properties were measured in Ringer's solution by Gamry Potentiostat. The I_{CORR} and E_{CORR} values were estimated using Gamry Echem Software and Tafel's extrapolation method. Coated samples were immersed in SBF solution for different periods of time, viz., 1 second, 24hours, 72hours and 1week and then further I_{CORR} and E_{CORR} values were estimated in Ringer's solution. For coated samples Electrochemical Impedance Spectroscopy were also done. Different parameters like Rp, alpha, Wd of EIS were used to evaluate the effectiveness of the coatings. Comparison of corrosion resistance among the coated samples revealed a few interesting characteristics. While DCPD coated Stainless Steel showed considerable improvement in corrosion resistance compared to as received sample, dip coated samples did not show appreciable improvement. Coated 316L shows better corrosion resistance than 316LN. Dip coated 316LN shows better corrosion resistance than 316L. So Electrophoretic Deposition gave much better coating in comparison to Dip coating. Coated samples were further studied by The Scanning Electron Microscope and Energy Dispersive X-Ray Spectroscopy. While SEM was done to ascertain uniformity of coating, EDAX was done to see the variation of calcium deposition as a function of different deposition parameters. Electrophoretic deposition gave much better coating and uniform variation of calcium compared to dip coating.

Keywords: Electrophoresis; Dipcoating; Ringer's solution

Introduction

During 1980s, biomedical implants were a feasible choice for patients undergoing joint replacement, but the orthopaedic market place failed to create cement less implant. As a consequence bio-active coatings were proposed as orthopedic implants. The biomedical industry used ceramic coating on stainless steel as they are wear-resistant and also aid in Osseo-integration between bone and implant [1]. The physical and mechanical properties analogous to device design and functionality are the added advantages of these Coatings. Electrophoretic deposition process is unique as the physical and mechanical properties of these coatings are the point of focus in medical implants [2]. Dip coating technology facilitates in designing an implant with beneficial mechanical properties and also provides superb synthetic bone properties [3].

In view of their processability, weldability, satisfactory mechanical properties, Metallic biomaterials are found to be beneficial in the field of biomedical materials. They degrade when in synergy with body fluids, which is the main drawback of these metallic biomaterials. Hence, care must be taken while using metallic biomaterials for conventional metallic implants. Corrosion resistance; the capacity to generate a protective passive film is checked before selecting the materials for implants. Commercially, 316L and 316LN stainless steels are widely used in biomedical applications. An external oxide layer protects the materials, assuring a satisfactory corrosion resistance. The trouble associated with corrosion is, release of ions from metallic species, that are harmful to the organism [4]. This protective passive layer decreases the corrosion rate and also ceases the ion release. Corrosion behavior of 316L and 316LN is the determining factor for their success as biomaterials [5]. The primary step for developing new biomaterials is, evaluation of their corrosion parameters in vitro. Among the mechanical properties, UTS (Ultimate Tensile Strength) is mainly concerned. UTS of 316L is 558Mpa and 316LN is 685Mpa [6].

In this study various experiments were done to test whether 316L and 316LN steel can be used as orthopedic implant. The 316L and 316LN were coated with Dicalcium phosphate di-hydrate by Electrophoretic deposition method and with polyvinyl alcohol by Dip coating method. The corrosion behavior of these two coated 316L and 316LN stainless steel were evaluated by electrochemical techniques. OCP (Octacalcium phosphate) and DCPD (Dicalcium phosphate di-hydrate) were deposited on Ti by ECR, whereas HAp was deposited on 316L alloy by EPD method [7]. So in this study DCPD has been deposited by electrophoresis. Samples were electrophoretically coated with Di-calcium phosphate di-hydrate, and dip coated with polyvinyl alcohol. Comparison of corrosion resistance among the coated samples revealed interesting characteristics. Coated 316LN showed better corrosion resistance than 316L. Dip coated 316LN shows better corrosion resistance than 316L. Coated samples were further studied by The Scanning Electron Microscope and Energy Dispersive X-Ray Spectroscopy. Though electrophoretic deposition gave much better coating and uniform variation of calcium compared to dip coating, E_{CORR}, I_{CORR} values of dip coated samples in Ringers's solution were better presumably because of formation of passive layer during dip coating. However stability of dip coated surface was poor.

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Received October 06, 2012; **Accepted** November 28, 2012; **Published** December 06, 2012

Citation: Ghosh S, Mitra PK (2013) Effectiveness of Dicalcium Phosphate Dihydrate as Biocompatible Coatings on 316L and 316LN Stainless Steel. J Material Sci Eng 2:118. doi:10.4172/2169-0022.1000118

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Experimental Procedure

Compositions of the samples are given in table 1.

Coating procedure

Two types of coatings were done**:**

Calcification with electrophoretic deposition: For EPD Dicalcium phosphate dihydrate ($CaHPO_4$,$2H_2O$) was used. It is practically insoluble in water, with a solubility of 0.02 g per 100 ml at 25°C. The sample to be coated was properly polished. Then the sample was used as anode and graphite plate was used as cathode. EPD experiments were done by varying the current, time, concentration of DCPD and voltage. The parameters are given in table 2. Calcification experiment was conducted by immersing the 316L and 316LN substrates in a phosphate-buffered solution, prepared by 8 gm Disodium hydrogen phosphate (Na_2HPO_4) and 0.1(M) HCl at around neutral pH at room temperature. The solution was then adjusted to slight super saturation with respect to Dicalcium Phosphate Di-Hydrate (DCPD), which potentially promotes the nucleation and calcification of the calcium phosphate crystals. After the calcium phosphate was deposited on the substrates, the substrates were washed with double distilled water and dried.

Dip coating procedure: For dip coating polyvinyl alcohol was used. After proper polishing, samples were dipped in a phosphate-buffer solution added with polyvinyl alcohol for three different periods of time, viz., 30 minutes, 45 minutes and 60 minutes. After the deposition, substrates were dried.

Stability of DCPD deposit in SBF solution: 316L and 316LN substrates after calcification were immersed in SBF at 37°C for 1second, 24hours, 72hours and 1week. After the immersion, the comparison of E_{CORR}, I_{CORR} values in SBF solution of the two different samples immersed for different periods of time were estimated.

Corrosion testing

Standard Electrochemical Corrosion Cell was used to perform the electrochemical potentiostatic polarization tests on standard flat metal specimens. Polarization experiments were carried out as per ASTM ST72 using Gamry Potentiostat. The software used was Gamry Echem Analyst. Potentiodynamic experiment in Ringer's solution with a scan rate of 1mV/sec was done with the as received, DCPD coated, Dip coated, and SBF solution immersed samples. I_{CORR} –E_{CORR} values were estimated from the polarization curves by Tafel's extrapolation method and are given in table 3 and a few typical polarization diagrams in Ringer's solution are given in figures 1-7. The composition of 250ml Ringer's solution is: NaCl -2.15gm/l, $CaCl_{2-}$ 0.0825gm/l and KCl -0.0759gm/l with pH 7.4 maintained throughout the experiment The Electrochemical impedance spectroscopy was done with the coated samples. Then the Bode plot, Nyquist plot and Rp and Ru values were obtained and the generated data from these curves are presented in table 3.

Results and Discussions

DCPD Coated Polarization curve (in Ringer's solution) (Figures 1-6).

Dip Coating polarization curves (in Ringer's solution) (Figure 7).

Stability test at SBF solution curves (Figures 8,9).

Polarization behavior of DCPD coated 316L and 316LN samples

There were four variable in the electrophoretic depositions, viz., time, current, voltage, and concentration. Polarization study revealed a definite pattern of these variables on the I_{CORR} and E_{CORR} values. The pattern of variation was different for 316L and 316LN.

Effect on 316L: With increasing time of deposition E_{CORR} becomes nobler but I_{CORR} remains almost same. At 60mins E_{CORR} is of -420mV vs. SCE and I_{CORR} is of .1µA/cm^2. It gives good passivity. With increasing current E_{CORR} becomes nobler and I_{CORR} keeps on decreasing. At 60mA current, E_{CORR} is of -462mV vs SCE and ICORR are of 1 µA/cm^2. This combination gives good passivity. In case of voltage variation, Figure 1 shows 5volt curve gives the noblest E_{CORR} i.e. -242mV vs. SCE and minimum I_{CORR} i.e. 9µA/cm^2. It shows the best passivity. At 10volt E_{CORR} and I_{CORR} are not up to the mark. At 2 volts E_{CORR} is almost same as

Sample	C	Mn	Si	P	S	Cr	Ni		Fe
316L	0.03%	2%	0.75%	0.03%	0.03%	18%	12%	2-4% (Mo)	Balance
316LN	0.08%	2%	0.75%	0.045%	0.03%	18-20%	10.5%	0.08% (N)	Balance

Table 1: Compositions of the samples.

Concentration	Concentration	0.02 g	0.06 g	0.075 g
	Voltage	500 volt	500 volt	500 volt
	Current	100 mA	100 mA	100 mA
	Time	30 min	30 min	30 min
Time	Time	45 min	60 min	1.5 hour
	Voltage	500 volt	500 volt	500 volt
	Current	50 mA	50 mA	50 mA
	Concentration	0.06 g	0.06 g	0.06 g
Current	Current	50 mA	60 mA	90 mA
	Voltage	500 volt	500 volt	500 volt
	Time	45 min	45 min	45 min
	Concentration	0.06 g	0.06 g	0.06 g
Voltage	Voltage	2 volt	5 volt	10 volt
	Current	500 mA	500 mA	500 mA
	Time	30 min	45 min	60 min
	Concentration	0.06 g	0.06 g	0.06 g

Table 2: Different parameter for DCPD Coating on 316L and 316LN.

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5volt. Concentration variation shows at 0.02g concentration E_{CORR} is of -900mV vs. SCE and I_{CORR} is of 90 µA/cm^2. It also shows good passivity. At 0.06g concentration E_{CORR} and I_{CORR} are not up to the mark. From the discussion of the effect of different parameters of Electrophoretic deposition on 316L it appeared that carrying out the deposition at 5 Volts with maximum current with 0.06 g concentration for 45 minutes would give the best coating. So the deposition was carried out with these parameters and polarization tests were done in Ringers solution. Figure 6 shows that E_{CORR} is of -242mV vs SCE and I_{CORR} is of 0.9 µA/cm^2. Interestingly this is the best corrosion resistance as was thought.

Sample	Variation		E_{CORR}	I_{CORR}
316L Stainless steel	as received		-300 mV	0.2 µA/cm^2
316LN Stainless steel	as received		-342 mV	0.1 µA/cm^2
316L	Concentration	0.02 g	-900 mV	90 µA/cm^2
		0.06 g	-850 mV	25 µA/cm^2
		0.075 g	-980 mV	95 µA/cm^2
	Time	45 min	-455 mV	8 µA/cm^2
		60 min	-420 mV	0.1 µA/cm^2
		1.5 hr	-415 mV	2 µA/cm^2
	Current	50 mA	-619 mV	9 µA/cm^2
		60 mA	-462 mV	1 µA/cm^2
		90 mA	-379 mV	1 µA/cm^2
	Voltage	2 volt	-264 mV	2 µA/cm^2
		5 volt	-242 mV	0.9 µA/cm^2
		10 volt	-435 mV	3 µA/cm^2
		316L best combination 5 volt	-242mV	0.9 µA/cm^2
316LN	Concentration	0.02 g	-450 mV	1.5 µA/cm^2
		0.06 g	-410 mV	1 µA/cm^2
		0.075 g	-800 mV	35 µA/cm^2
	Time	45 min	-205 mV	0.2 µA/cm^2
		60 min	-255 mV	0.2 µA/cm^2
		1.5 hr	-244 mV	0.7 µA/cm^2
	Current	50 mA	-271.4 mV	0.65 µA/cm^2
		60 mA	-171.4 mV	0.45 µA/cm^2
		90 mA	-235.7 mV	0.7 µA/cm^2
	Voltage	3 volt	-280 mV	0.6 µA/cm^2
		5 volt	-750 mV	0.7µA/cm^2
		10 volt	-900 mV	0.9 µA/cm^2
		316LN best combination 60 mA	-171.4 mV	0.45 µA/cm^2

Table 3: Estimation of I_{CORR} –E_{CORR} values from the polarization curves by Tafel's extrapolation method.

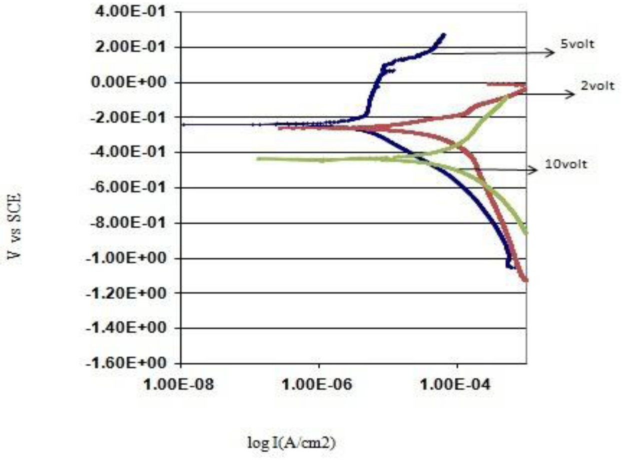

Figure 1: 316L DCPD coating at different voltages curve.

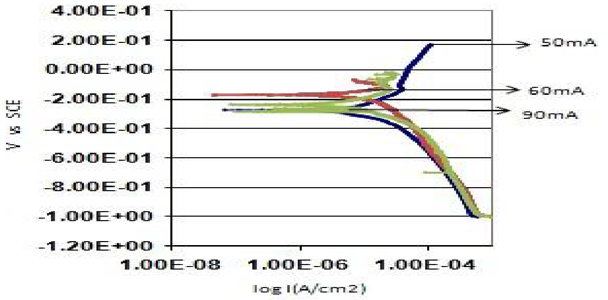

Figure 2: 316LN DCPD coating at different current curve.

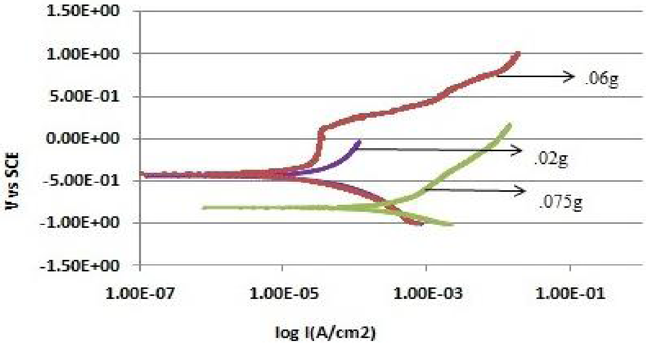

Figure 3: 316LN DCPD coating at different concentrations curve.

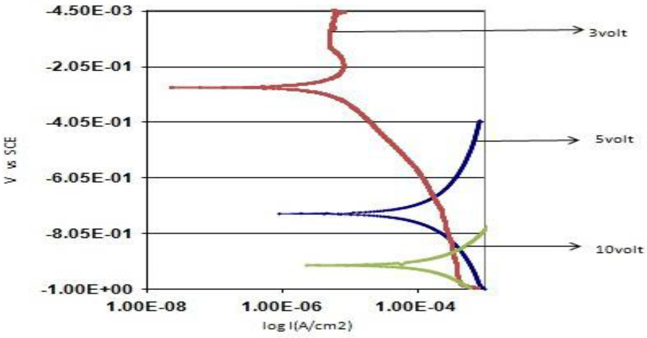

Figure 4: 316LN DCPD coating at different voltage curve.

Effect of 316LN: With the increasing time E_{CORR} becomes active. At 45 mins the passivity is appreciable. At 45 mins (Figure 5) E_{CORR} is of -205 mV vs SCE and I_{CORR} is of .2 µA/cm^2. In case of current variation, figure 2 shows at 60 mA, the noblest E_{CORR} is of -171.4 mV vs SCE and almost same range of I_{CORR} is of 0.45 µA/cm^2 for all. At 60 mA curve tends to show the best passivity. At 3 volt (Figure 4) E_{CORR} is of -280 mV vs SCE and I_{CORR} are of 0.6 µA/cm^2 and it gives good passivity. With increasing voltage all these become poorer. Figure 3 shows with increasing concentration E_{CORR} becomes nobler and I_{CORR} decreases. Passivity also increases with the increasing concentration. At 0.075 g

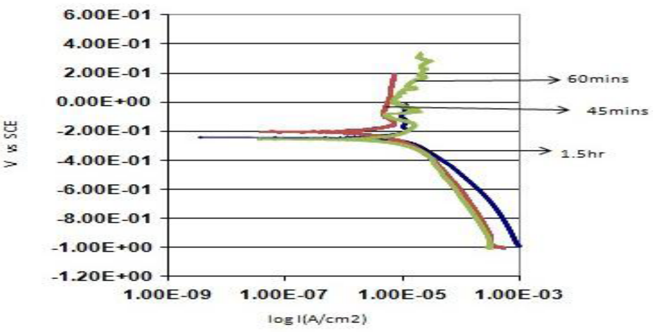

Figure 5: 316LN DCPD coating at different time curve.

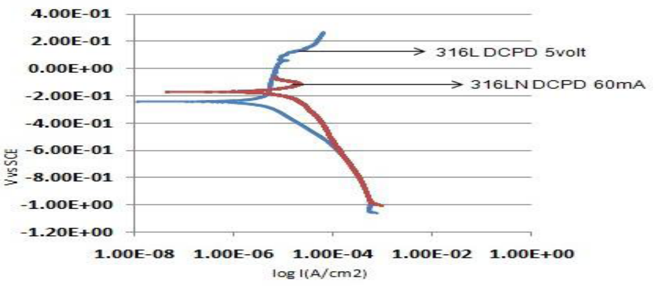

Figure 6: 316L-316LN DCPD best combination curve.

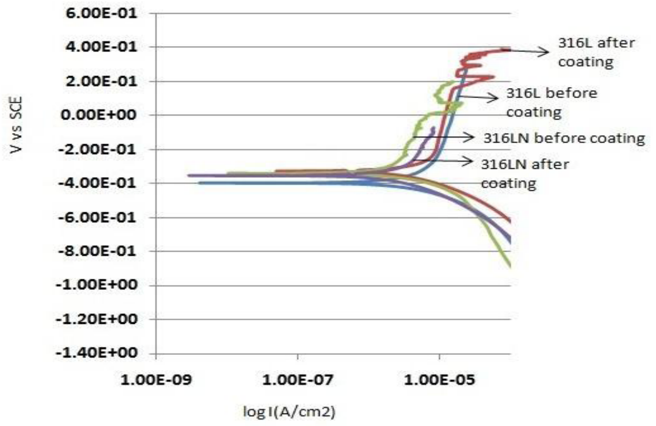

Figure 7: 316L-316LN before-after coating curve.

concentration E_{CORR} is of -800 mV vs SCE and I_{CORR} is of 35 μA/cm^2. Similarly for 316LN it appeared that carrying out the deposition at 60 mA and .06g concentration with maximum current for 45 minutes would give the best coating. So the deposition was carried out with these parameters and polarization tests were done in Ringers solution. Figure 6 shows E_{CORR} (-171.4 mV vs. SCE) and I_{CORR} (0.45 μA/cm^2). Interestingly this too reflects the best corrosion resistance. Moreover temperature also plays a very important role (Tables 3-5). The deposition experiments were carried out over the period of September to May at room temperature, which varied substantially.

Stability of the DCPD coated 316L and 316LN samples in SBF solution

SBF (pH 7.25) [8] is a metastable solution containing calcium and phosphate ions already supersaturated with respect to the apatite. In case of 316L from the figure 8 at different time periods (1second, 24hours, 72hours, 1week) it can be seen that for all immersion times, i.e., for 1 second, 24 hours and 72 hours of immersion give almost same E_{CORR} and I_{CORR}. But 1week curve gives much active E_{CORR} of -631 mV vs SCE and much higher I_{CORR} of 200 μA/cm^2. Beyond particular time period stability decreases. Figure 9 shows the corresponding behavior for 316LN at 1second, 24 hours, 72 hours and 1 week time periods. E_{CORR} and I_{CORR} vary sinusoidally. The stability of 316LN is found to be better than that of 316L.

Polarization behavior of Dip coated 316L and 316LN samples

In case of 316L, figure 7 shows, dip coated sample at 45 mins gives noblest E_{CORR} of -328.1 mV vs. SCE and minimum I_{CORR} of .3 μA/cm^2 in comparison with DCPD coating. In case of 316LN, figure 7 shows dip coated sample with active E_{CORR} of -351 mV vs. SCE and minimum I_{CORR} of .07 μA/cm^2 which is also better with respect to DCPD coated sample. Though dip coated samples exhibit much superior corrosion resistance I_{CORR} in the nano range but on immersing the dipcoated sample in SBF coats come off (Table 6).

Analysis of EIS data

EIS study of a few selected coated samples was done. The study revealed CPE with Diffusion. Barring a few in most of the cases fits were good. This suggests very effective coating or formation of continuous layer over the surface during coating. Even the dip coated samples which showed good corrosion resistance shows good fit suggesting that corrosion resistance obtained maybe due to the inherent nature of the alloy. In case of 316L DCPD coated at 5volt gives best result interms of higher Ru (uncompensated resistance) i.e. 34.67 ohms and Rp value is

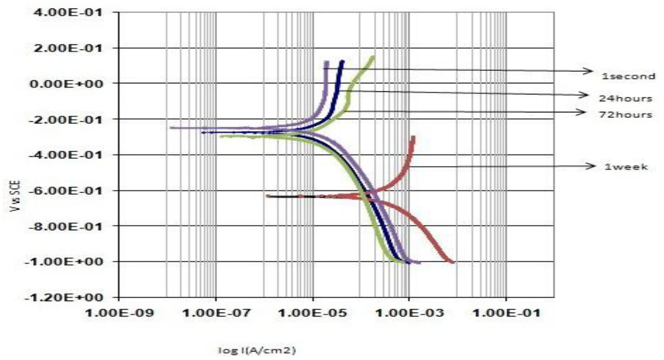

Figure 8: 316L DCPD stability test curve.

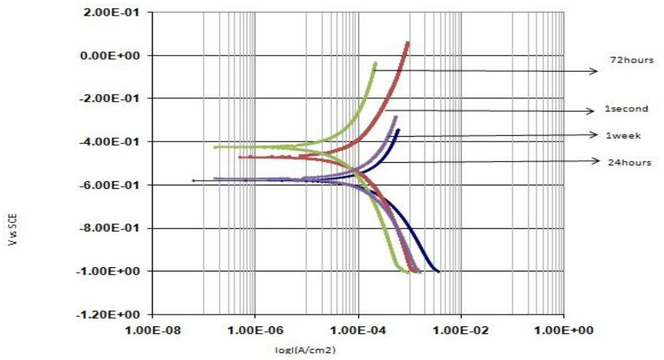

Figure 9: 316LN DCPD stability test curve.

Sample	Time	E_{CORR}	I_{CORR}
316L	1second	-245 mV	3 μA/cm^2
	24hours	-271 mV	3 μA/cm^2
	72hours	-294 mV	4 μA/cm^2
	1week	-631 mV	200 μA/cm^2
316LN	1second	-471 mV	40 μA/cm^2
	24hours	-584 mV	40 μA/cm^2
	72hours	-420 mV	20 μA/cm^2
	1week	-590 mV	40 μA/cm^2

Table 4: Corrosion rates of samples immersed into SBF solution.

Sample	E_{CORR}	I_{CORR}
316L 45mins	-328.1 mV	0.3 μA/cm^2
316LN 45mins	-351 mV	0.07 μA/cm^2
316LN 30mins	-773.8 mV	0.5 μA/cm^2

Table 5: Corrosion rates of Dip coated samples.

Sample	Ru (ohms)	Y_0 (s*s^a)	alpha	Wd (s*s^(1/2))	Rp (ohms)
316L DCPD 5volt	34.67	269.2e-6	658.1e-3	1.400e-9	18.04
316LN DCPD 2volt	33.22	47.67e-6	762.4e-3	130.8e-6	1.179e3
316L DCPD 90 mA current	34.14	82.31e-6	628.0e-3	175.0e-6	7.894e3
316LN DCPD 60 mA current	73.75	50.53e-6	565.3e-3	86.54e-6	8.001e3
316L DCPD 60mA current	48.28	56.54e-6	625.4e-3	105.7e-6	3.618e3
316LN Dip coating 45min	40.40	67.95e-6	624.3e-3	15.27e-6	5.459e3
316L Dip coating 45min	38.49	63.59e-6	621.3e-3	78.17e-6	1.305e3
316LN DCPD 50mA current	539.8e-3	328.4e-9	176.2e-6	39.29e-9	513.8e3
316LN DCPD 0.06g	2.227e-9	23.93e-6	358.3e-3	155.3e-6	96.21
316L DCPD 0.075g	1.224e-9	7.047e-9	1.000	354.7e-6	20.49
316LN DCPD 0.075g	0.043e-9	2.682e-9	999.90e-3	126.6e-6	28.11

Table 6: EIS data with CPE with Diffusion Model.

Sample	Na K_α	P K_α	Ca K_α	Cr K_α	Fe K_α	Ni K_α	O	Mo L_α	N K_α	Cl K_α
316L DCPD (60mA) POS1	2.94	1.55	.67	9.23	28.47	4.41	52.73			
316L DCPD (60mA) POS2		.53	.79	8.77	30.66	6.65	52.59			
316L DCPD (60mA) Average		.96	.65	8.05	28.79	3.81	55.24	2.50		
316LN DCPD (60mA) POS1	2.23	.49	.30	3.84	33.53	.04	62.64	.60	15.28	.40
316LN DCPD (60mA) POS2		1.36	1.06	5.33	9.52	3.00	63.83	.48	14.97	.46
316LN DCPD (60mA) Average	2.92	.98	.09	3.45	10.48	3.01	62.44	.76		.86
316L Dipcoating(45min) POS1	2.72	1.18		15.19	49.08	8.45				
316L Dipcoating(45min) POS2	4.98	1.55		14.53	45.67	7.81	17.98			.26
316L Dipcoating(45min) Average	.07	.47		18.28	61.42	10.99	6.94			
316LN Dipcoating(45min) POS1	13.48	4.71		8.63	31.82	4.78	30.35			.37
316LN Dipcoating(45min) POS2	7.99	.51		14.39	49.26	8.63	15.48			.07

Table 7: Comparative study of EDX (Atomic %) of DCPD and Dip coated samples.

18.04 ohms. In case of 316LN DCPD coated sample 60mA gives best result interms of higher Ru i.e. 73.75 ohms. As we all know higher the value of Ru and Rp higher the stability of coating (Table 7).

SEM of 316L SS DCPD coated samples

Scanning Electron Microscopic and EDX study of DCPD and Dip coated 316L and 316LN samples: Morphology of the coatings was investigated by scanning electron microscopy with associated energy dispersive spectroscopy analysis (SEM-EDS). Figure 10 shows coating morphology of 316L DCPD coated samples at x10,000 magnification. The coating is uniform and there are no cracks. The satisfactory adhesion between the coating and substrate suggests its suitability for load-bearing capability. Figure 11 shows coating surface of 316LN sample, which shows better uniformity in coating than 316L at the same magnification. Figure 12 shows dip coated surface of 316L at x200 and x500 magnification. It shows porosity in coated surface. Figure 13 shows layered structure of 316LN dip coated surfaces at x1,000 and x2,000 magnifications. Since these specimens exhibited very good corrosion resistance and passivity, these layers are presumably oxide (passive) layers. The comparative EDX data (atomic%) of different coated samples is given in

DCPD coating equation:

$$CaHPO_4{\cdot}2H_2O + Na_2HPO_4 + 2HCl \rightarrow Ca(H_2PO_4)_2 + 2NaOCl + 2H_2 \quad (1)$$

Here from the equation it can be seen that Di-calcium phosphate dihydrate reacts with Di-sodium hydrogen phosphate and Hydrochloric acid and produces Mono-calcium hydrogen phosphate (anhydrous MCPA) [2,6], and Sodium hypo-chloride. From the products of the

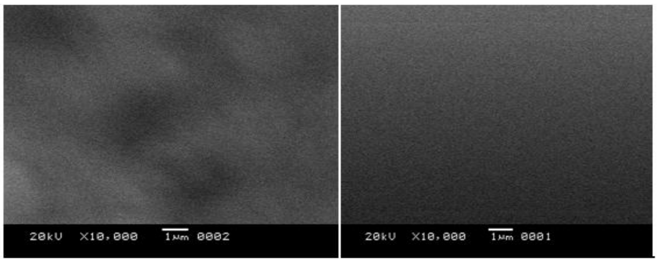

Figure 10: SEM images of 316L SS DCPD (at 60 mA) coated sample.

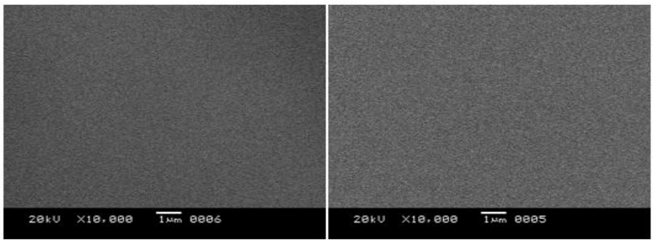

Figure 11: SEM images of 316LN SS DCPD (at 60 mA) coated sample.

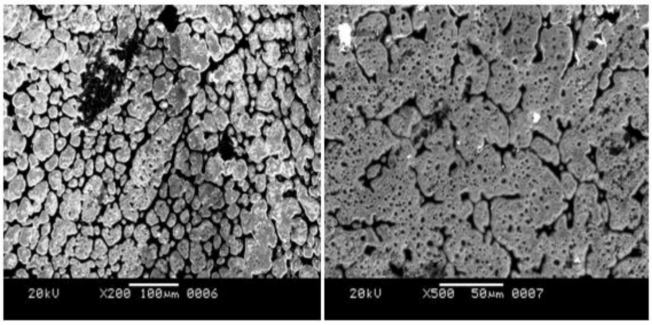

Figure 12: SEM of 316L SS Dip coated (45minutes).

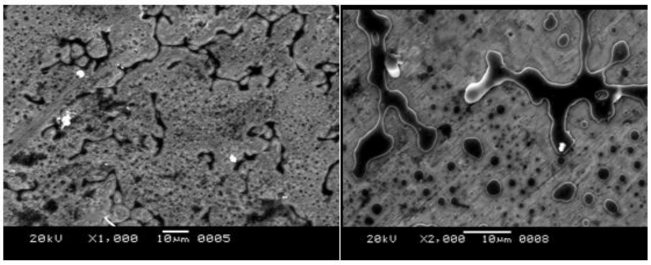

Figure 13: SEM of 316LN SS Dip coated (45minutes).

above equation it can be seen stoichiometrically Calcium ratio is half of the phosphorous. In case of DCPD coated (at 60mA current) 316L sample, position 1 shows PK_α is 1.55 atomic% and CaK_α is .67 atomic%, which satisfies the coating equation stoichiometricaly. DCPD coated (at 60mA current) 316L sample, position 2 shows PK_α is .53 atomic% and CaK_α is .79 atomic%, which somehow differs the equation. From the above discussion it is apparent that by Electrophoretic route it is mono calcium phosphate (anhydrous) that gets deposited rather than Dicalcium phosphate dihydrate.

Dip coating equation:

The possible reaction that takes place during dip coating is given below

$$(C_2H_4O)_n + Na_2HPO_4 + 2HCl \rightarrow \text{coated material} + H_2O \qquad (2)$$

There is OH^- group associated with PVA .So there is some unsaturated charge with this oxygen of this OH^- group which can form bond either with metal oxide or metal. For elements with unsaturated 3-D orbits the bond is quite stronger. So it is expected it will form either metal hydroxide or Me-O hydroxide with 316L and 316LN SS. This acts as passive layer and may improves the corrosion resistance of the dip coated SS. In case 316L, dip coating at 45 mins (position2) CrK_α is 14.53 atomic% and O is 17.98 atomic% .This indicates formation of Cr_2O_3. Presence of chlorine can also be seen here. However, average oxygen is lower. In case of 316LN dip-coating at 45mins (position1) shows 30.35 atomic% of O. But in case of 316LN at position 2 there is a mere presence of chlorine.

The EDX data corroborates the SEM finding that oxygen content in the dip coated specimens are higher. So the inference drawn earlier to explain better polarization behavior of dip coated samples the passivity is the cause for better corrosion resistance is substantiated by the SEM-EDX study.

Conclusion

1. 316LN showed greater improvement in corrosion resistance property than 316L and also 316LN showed better passivity after coating.
2. Electrophoretic deposition of DCPD could be done though improvement in corrosion resistance was not up to a high level.
3. Dip coated samples showed remarkable improvement corrosion resistance property and passivity.
4. DCPD deposition was more uniform than dip coating (PVA).
5. EIS experiments showed CPE with Diffusion model for DCPD coated samples indicating reasonable coating.
6. By Electrophoretic deposition Mono-calcium phosphate (anhydrous MCPA) is obtained.

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