Evolution on the Microstructure of Alumina Coated Stainless Steel Using Electro Phoretic Deposition

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***Abstract*-This paper emphasizes on the microstructure of Al_2O_3 coating fabricated on biomedical 316L stainless steel using Electrophoretic deposition technique (EPD) by varying the voltage from 10-70 V. An adherent dense coating was obtained at 55 V. The microstructure of the coated surfaces was characterized using Scanning electron microscope (SEM). The coatings fabricated at 55 V have resulted in higher adhesion, lower porosity as compared to the bare substrate.**

***Key words:* Al_2O_3, Microstructure, EPD**

I. INTRODUCTION

EPD has received increasing attention because of its important advantages such as low equipment cost and starting material, possibility to realize unique microstructures and nanostructures as well as novel and complex materials combinations using complex substrates. Many different materials have been deposited by EPD, like ceramic materials [1, 2], functionally graded materials [3, 4], polymers [5] and combinations of different materials [6, 7] for important applications such as optics, thin films, fuel cells, high performance ceramic and composite coatings , biomaterials and electrochemical capacitors. Electrophoretic deposition is a process in which colloidal particles dispersed in a liquid medium migrate toward the electrode with the opposite charge under the influence of an electric field (electrophoresis) and are deposited on the substrates. In EPD, organic solvents and/or water are used. Water is used as solvent for EPD mainly because of higher-temperature control during the process or a faster kinetics (because of a high dielectric constant), cost benefits [8, 9], and is beneficial in preparing stable suspensions of oxide powders. However, the electrolysis of water that accompanies EPD interferes with the quality of the deposits at high applied voltage [10, 11]. Even at low voltages, electrolysis of water occurs, and gas evolution at the electrodes is inevitable at field strengths high enough to give reasonably good deposits. The electrochemical reaction in the electrodes when current is passed through affects the efficiency of the process and the uniformity of the deposits.

Several authors have conducted electrophoretic deposition in a variety of different organic solvents. For example, Anné et al. [12] evaluated ethanol solvent and mixture of methylethylketone and butyl amine on the basis of electrophoretic yield rate and the electric field as a function of deposition time during EPD of Al_2O_3.They revealed that suspension composition determines whether a potential drop is generated at the deposition electrode or not and an electrical resistive layer was formed in ethanol solvent with certain additives. Novak and Konig [13] compared electrophoretic deposition of alumina powder in ethanolic and in aqueous suspensions, and related that water suspensions appear to be advantageous over ethanol, from standpoint of higher achievable green and sintered densities of the deposits and higher deposition rates. As shown by examples above, Al_2O_3 based coatings have been intensively studied due to their potential applications in solar absorbers [14], thin film, oxide fuel cell electrode [15,16], and electrochemical capacitors [17]. Hence an attempt has been made to deposit Alumina over Stainless Steel subtract using Electro Phoretic Deposition.

II. EXPERIMENTAL

- *Materials and Slurry Preparation*

The calcined alumina powder (AP001 grade) procured from CHENNAI METCO, INDIA, is used for the Electro Phoretic Deposition process. The powder has a mean particle size of 0.7μm. All the coatings were performed on 6 specimens of 316L grade stainless steel of dimensions 20x20x2.5mm and surface are of $20mm^2$. The aqueous slurries with different solid loadings (5, 10, 15, 20% wt) were used in preparations The organic solvents like ethanol, methanol was mixed with deionized water at various proportions. The deionized water is used as the dominating solvent in order to increase the physiochemical properties of the slurries. To stabilize slurries various dispersant such as EDTA, acetic acid, nitric acid, dextrin, calgon, were used at different concentrations (0-5%wt). The parameters varied to optimize the slurry are voltage, current, deposition time, pH, and distance between the electrodes. Thus the slurry characterization is done by varying the above parameters. The optimized parameters to prepare the slurry and to execute EPD process, obtained from the experimental work are listed in table 1.

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Table 1

Parameters	Suspension composition
Dominating solvent	Deionized Water (80, 70, 60 Vol %)
Organic solvent	Ethanol/Methanol (20, 30, 40 Vol %
Dispersant	Acetic Acid
Type of EPD	Cathodic EPD
Solid Loading (Al_2O_3-(g))	5
pH	2-5
Temperature (^{0}c)	24
Distance between the electrodes (cm)	1
Voltage (v)	55
Current (A)	1
Deposition time (min)	1

The parameters mentioned in table 1 were kept constant for all the coatings obtained from the experimental work

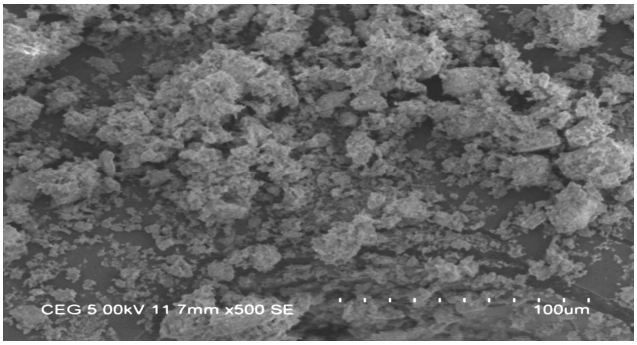

Fig1. SEM – Al_2O_3 Powder

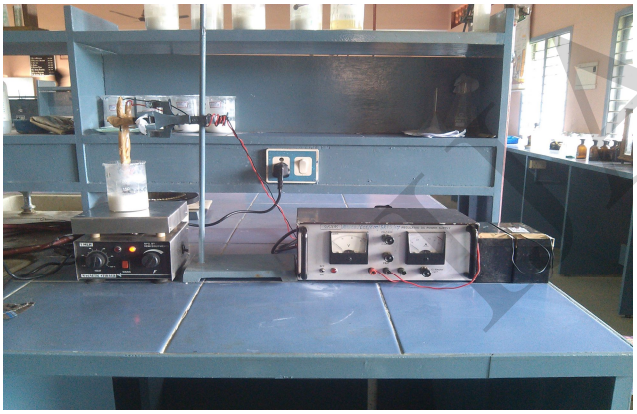

Fig2. Electro Phoretic Deposition Set up

- *Powder and Coating Characterization*

The samples were polished using the emery paper of different grit size ranging from 220 µ m - 1000µ m The morphologies of the starting powder and the coatings were observed by JE0L.JSM-6360 Scanning electron microscope (SEM), for surface morphological and cross sectional studies..

III. RESULTS AND DISCUSSSION

- *Surface Morphological Analysis*

The surface morphologies of the Al_2O_3 coated surfaces were observed for all the coated specimens to consist, fine particles, pores, cracks. The morphological study of the surface of the coating, at higher magnification reveals a large amount of particles at agglomerated state. The porosity was observed to be less in all the coatings and no cracks were seen during the analysis, because this is a aqueous based coating method processed at low temperatures, hence the particles remain as un-melted, there is no radial cooling effect takes place so no cracks were observed on drying the material. The coatings obtained were dense but not uniform for all the coatings, which is consistent to the previous researchers [18]. The particle size was not able to be determined after coating, due to agglomeration of the particles. It is noteworthy to state that the particle size has increased as studied in other researches [19].

On comparing the surface morphologies of the coatings obtained the methanol content slurries resulted in effective deposition when compared with the ethanol content slurries, since methanol has high relative dielectric constant and conductivity [18-21]. Thick and dense coatings were obtained at (80/20, 70/30)-proportions of deionized water and organic solvents. The porosity seems to be less in case of (60/40) – proportion.

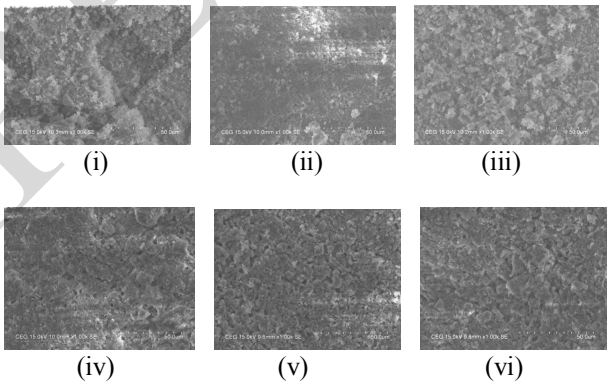

Fig3. Surface Morphologies of Al_2O_3 Coatings
(i) 60% deionized water 40% ethanol(ii) 70% deionized water 30% ethanol(iii) 80% deionized water 20% ethanol
(iv) 60% deionized water 40 methano (v) 70% deionized water 30% methanol(vi) 80% deionized water 20% methanol

- *Cross Sectional Analysis*

The cross sections of the coatings were observed to consist of adhesion strength, cracks, pores, and thickness of the coating. Better adhesion was noticed between the substrate and the coating. The amount of deposited alumina varied from 2g-5g. Since the EPD process was carried out at low temperature the porosity was observed to be less and no cracks were seen on the coated surface. The thickness of the coatings was found to vary from 50µm-100µm. The particles were observed to be agglomerated. Dense, non-uniform coating was observed, an efficient and effective deposition is observed at all coatings when compared to previous researchers [21].

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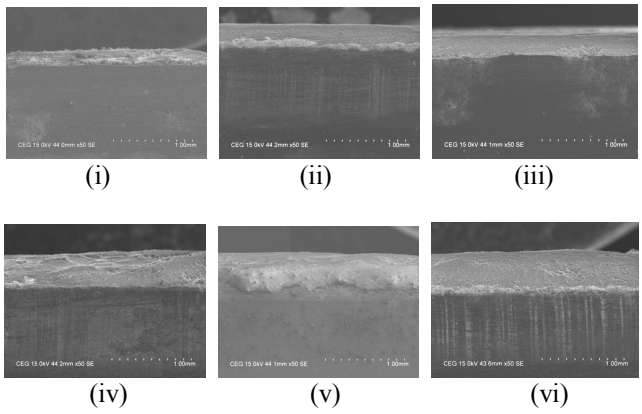

Fig4. Cross Sectional Morphologies of Al_2O_3 Coatings
(i) 60% deionized water 40% ethanol. (ii) 70% deionized water 30% ethanol. (iii) 80% deionized water 20% ethanol (iv) 60% deionized water 40 methanol (v) 70% deionized water 30% methanol (vi) 80% deionized water 20% methanol

IV. CONCLUSION

The morphology of the electro phoretic deposited coating carried out at 55V has resulted in dense and uniform microstructure. Among all the coatings 60/40 ratio of organic solvents with deionized water has resulted in low porosity compared to other researchers. The cross section morphology reveals better Interfacial adhesive between the coating and subtract in all the coatings.

V. COMPETING INTERESTS

Authors have declared no competing interests exist.

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