

OPTIMIZATION OF PROCESS PARAMETERS FOR MICRO HARDNESS OF ELECTRODEPOSITED TiO₂-HAP COATING ON Ti-6Al-4V SUSTRATE USING TAGUCHI METHOD

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Abstract— Aim of this work is to fabricate TiO₂-HAP composite coatings on Ti-6Al-4V alloy with electrodeposition process and examine the effect of process parameters on micro hardness of TiO₂-HAP composite coated Ti-6Al-4V substrate. Voltage, pH of electrolyte and Titania (TiO₂) concentration were taken as process parameters. TiO₂-HAP composite coatings on Ti-6Al-4V were deposited with help of electrodeposition process from the standard Watt's solution. The relationship between process parameters and micro hardness of composite coatings was established by Taguchi method. Design of experiment was carried out by selecting three levels of each parameters and L9 Taguchi orthogonal array design was employed for this purpose. Vickers micro-hardness tester was used to determine micro-hardness of the composite coatings by applying 200 gf for 15 seconds of indentation period. Signal-to-noise ratio and analysis of variance were employed to determine the significance of the process parameter. The surface morphologies and elemental compositions of coating were analyzed by using scanning electron microscope and energy dispersive spectroscopy.

Keywords—composite coating, electrodeposition, hydroxyapatite, hardness, orthogonal array

I. INTRODUCTION

Due to excellent oxide film formation capability on surface and superior biocompatibility, titanium alloys are extensively used in biomedical applications such as orthopaedic and dental implants [1]. However, failure of an implant can occur due to insufficient bone-implant contact, dissolution of metal ions on tissues adjoining surface of implant and formation of wear debris due to joint movements which leads to biodegradation of implant [2–3]. Due to high chemical binding capability to living bone and excellent mechanical properties, hydroxyapatite (HAP, Ca₁₀(PO₄)₆(OH)₂) coating on metal substrates (e.g., Co alloy, Ti alloy or stainless steel) has drawn considerable attention to achieve superior bioactivity [4]. Hydroxyapatite coatings have been developed on the substrate surface by various methods and techniques which include chemical vapour deposition (CVD), plasma spray method (PSM), physical vapour deposition (PVD), sol-gel technique, ion implantation, and Electrodeposition [5]. However Electro deposition is the most suitable coating technique due to various advantages linked with the process such as low cost and low energy requirement, feasibility to coat complex shaped parts and control over the process parameters during the deposition [6]. Specific surface properties can be imparted to substrate surface such as hardness and adhesion between coating and metal surface [7]. The elements with requisite characteristic can be implanted in matrix material through electrodeposition process to produce composite coating [8]. The reinforcing elements embedded in the composite coating contain ceramic particles (TiO₂, Si₃N₄, Al₂O₃, SiC, TiN, and ZrO₂), metal particles (W, Cu, V, Ti, Cr, Mo and Al), diamond particles, PTFE, pumice and carbon fibres [9]. Electrodeposition process can also be employed to produce composite coatings under controlled conditions of process parameters. The various process parameters which influence the process are current applied, voltage, pH of electrolyte, particle concentrations, bath temperature and agitation of electrolyte solution [10].

HAP based composite coatings have been developed by various researchers. Qiu *et al.* [11] deposited HAP-TiO₂ composite coatings on the surface of Ni-Ti alloy by electrodeposition process. Addition of TiO₂ to the electrolyte results in needle-flower-like crystals of HAP. Adhesive strength of HAP-TiO₂ coating and HAP coating were measured to be 23.7MPa and 13.4MPa respectively. Thicknesses of coatings were 7µm in both cases. Qiu *et al.* [12] also produced HAP-ZrO₂ composite coatings on Ni-Ti alloy by the same process. The needle-flower-like crystals of HAP were obtained due to addition of ZrO₂ to the electrolyte solution. HAP-ZrO₂ coated samples exhibited almost 60 times lower corrosion current density in comparison to uncoated sample. HAP crystal grains were arranged along the [0 0 1] direction in the coating. The thickness of the HAP and composite HAP-ZrO₂ coatings were about 7µm and 8µm respectively. The adhesive strength of HAP-ZrO₂ coating and HAP coating were measured to be 24.2 MPa and 13.4 MPa respectively. Lee [13] studied the effect of electroplated HAP-TiO₂ composite coatings on the wear and corrosion resistance of

Ti-6Al-4V alloy in Hanks' solution. Anodizing process was carried out at 10 V with different times of 40, 50, 60, 120 and 180 min respectively. After the anodizing process, constant current density of 10mA/cm² was used to fabricate HAP-TiO₂ composite coatings at 80 °C for 60 min. The electrolyte was magnetically agitated at a speed of 200 rpm to produce HAP-TiO₂ coating. Micro hardness of the coatings was ranging from 380.62 to 890.92 HV and the maximum hardness of 890.92HV was obtained with 180 minute anodized sample. R_a and RMS values varied from 500-650 nm and 750-900 nm respectively and higher values corresponds to higher duration of anodizing. When anodizing time for samples increased, decrease in coefficient of friction and increase in corrosion resistance and of the coatings were observed. **Huang et al. [14]** presented the antibacterial efficacy and cyto-compatibility of electroplated Cu and Zn co-substituted HAP (Zn-Cu-HAP) coating on CP-Ti. Cu²⁺ was substituted into HAP structure to improve antibacterial property. Cu ions released from Zn-Cu-HAP and produced strong antibacterial effect due to high antimicrobial ratio (K> 95%). Zn²⁺ acts as secondary material into Cu-HAP to reduce Cu concentration. A dense coating of thickness approximately 10µm was obtained. The adhesion strength of Zn-Cu-HAP and HAP coating was approximately 12MPa and 10MPa respectively. Zn-Cu-HAP coating exhibit superior corrosion protection for Ti substrates.

The effects of process parameters had been investigated by the numerous researchers using different techniques. Ni-Al composite coating was prepared by **Daemi et al. [15]** from Watt's solution of nickel. It has been observed that increase in current density enhanced the weight percent of Al particles in coated substrate. **Cai and Jiang [16]** reported that effective control can be exercised over current density and particle concentration for improvement of hardness of Ni-Al composite coating. The effect of micro and nano level particle size of Al on the structure of Ni-Al composite coating was investigated by **Zhou et al. [17]** by using the electro deposition process. Nano Al particles resulted in dense and homogeneous structure with large number of particles per unit volume in the composite matrix. A prediction model for copper content was developed by **Subramanian et al. [18]** in electrodeposition of bronze with help of neural network techniques. Ni-Diamond composite coating was developed by **Ramanathan et al. [19]** through design of experiments (DOE) approach and current density, pH and temperature of the electrolyte were taken as process parameters. ANN and regression modelling techniques were used to develop a prediction model for volume fraction of diamond particles. Ni/Ti, Ni/Al and Ni/Ti/Al composite coatings were prepared by **Naploszek et al. [20]** the with electrodeposition technique. The deposition of Ti and Al particles were obtained in Ni matrix. **Prakash et al. [21]** prepared the Ni- YSZ composite coatings and studied the effect of process parameters on the composite coating by L9 orthogonal array of Taguchi's design and analysis of S/N ratios. Particle concentration, current density, and deposition time were considered as process parameters and micro hardness, thickness of coating and area fraction of YSZ particles were considered as output responses. Mean S/N ratios of output responses were evaluated to determine significant parameters. AA7075-TiC metal matrix composites were developed by **Baskaran and Anandkrishnan [22]**. The dry sliding wear behaviour of composites was studied by designing the experiments using L27 orthogonal array of Taguchi technique. TiC (wt.%), load(N), sliding velocity (m/s) and sliding distance (m) were taken as process parameters. The load and sliding velocity were evaluated as highly significant parameters on the wear rate. The optimum levels of process parameters were evaluated with help of various statistical prediction models by many researchers [23-26].

In all electrodeposition procedures, current, voltage, pH of electrolyte, particle concentrations, electrolyte temperature and agitation of electrolyte were considered as process parameters. In this present work, a composite coating of TiO₂- HAP has been developed on Ti-6Al-4V substrate by electrodeposition by considering voltage, pH of electrolytic and titania concentration as process parameters. These process parameters are optimized for maximum micro hardness by using Taguchi methodology.

II. DESIGN OF EXPERIMENTS BY TAGUCHI METHOD

Taguchi technique employs design of experiments as one of the highly beneficial statistical tool which is used to examine the effect of variables and their interactions on the output of the process. Voltage, pH of electrolytic bath, electrolyte concentration, and current density, agitation of electrolyte, duty cycle, and temperature of electrolyte solution are the main process parameters. Out of these parameters, voltage, pH of electrolytic bath and titania concentration were selected as independent parameters for electrodeposition of TiO₂-HAP composite coatings on Ti-6Al-4V substrate. Three levels were selected for each of three independent parameters and a L9 orthogonal array was formed. The levels of parameters selected are shown in the Table I.

TABLE I VARIOUS PARAMETERS AND THEIR LEVELS

S.No.	Parameters	Level 1	Level 2	Level 3	Units
1	Voltage	1	1.5	2	Volt
2	pH Of Electrolyte	3.3	4.3	5.3	-
3	Titania Concentration	3	4	5	g/L

The signal-to-noise (S/N) ratio is determined to obtain optimum values of parameters at maximum response value. Three types of S/N ratio are used based on characteristic of output: lower is better, nominal is better and higher is better.

The characteristics of S/N ratio, ‘higher-the-better’ have been used to obtain the optimized parameters for higher micro hardness.

- (i) Response Name : Micro-hardness
 Response Type : Higher-the-better
 Units : HV (Vickers Hardness Number)

S/N ratio for “larger is better” is given by equation (1):

$$S/N = -10 \cdot \log \left(\frac{1}{n} \sum \frac{1}{y_i^2} \right) \quad \dots(1)$$

Where n is the no of tests and y_i is the value of experimental result of i^{th} test. After computation of all S/N ratios for each run of an experiment, a graphical approach is advised by Taguchi to analyze the data. The prepared Taguchi design for electrodeposition process is shown in Table II.

TABLE II TAGUCHI DESIGN FOR ELECTRODEPOSITION PROCESS

Exp. No.	Voltage (V)	pH of Electrolyte	Titania Concn. (g/L)
1	1	3.3	3
2	1	4.3	4
3	1	5.3	5
4	1.5	3.3	4
5	1.5	4.3	5
6	1.5	5.3	3
7	2	3.3	5
8	2	4.3	3
9	2	5.3	4

III. MATERIAL AND METHODS

A. Substrate Preparation

The rectangular shaped specimens of 2.5mm × 20mm × 25mm size were cut from the titanium plate on Electric Discharge Wire Cut Machine. The samples were grinded with emery papers of 80, 200, 400, 800, 1000 no. respectively to obtain a better surface finish. The substrate was rinsed in acetone for 24 hours before deposition and then washed with distilled water followed by drying in air.

B. Preparation of Electrolyte Solution

The electrolyte solution of specific concentration in aqueous media was prepared to deposit TiO₂-HAP on titanium substrate by electrodeposition process. The electrolyte solution was prepared by taking 0.042M Ca (NO₃)₂·4H₂O and 0.025 M NH₄H₂PO₄ concentration of constituent’s salts. To prepare TiO₂- HAP composite coatings, 3-5 g/L TiO₂ was added into the electrolyte. The pH of the electrolyte was maintained at 3.3 at 28 °C by adding nitric acid and ammonia as per need. Table III provides the composition of watt’s solution, as well as, the deposition conditions.

TABLE III WATT’S SOLUTION FOR TiO₂-HAP COATINGS

Electrolyte (Watt’s) Solution Composition	
Concentration of Ca (NO ₃) ₂ ·4H ₂ O, g/L	9.918
Concentration of NH ₄ H ₂ PO ₄ , g/L	2.875
Concentration of TiO ₂ , g/L	3-5
pH of electrolyte	3.3-5.3
Temperature, °C	30-50
Substrate	Ti-6Al-4V alloy
Voltage, V	1, 1.5, 2
Stirrer, rpm	701

C. Ultrasonic Agitation of Electrolyte

The electrolyte solution is ultrasonically agitated for 30 minutes at 50 KHz frequency to break up the salts particles into nano particles to obtain a saturated solution. Electrolyte beaker was place over the magnetic stirrer with temperature control to agitate the solution at 701 rpm along with heating up to 50⁰C.

D. Electrodeposition

An Auto lab Potentiostat, PGSTAT302N, three electrode electrochemical workstation was used to deposit TiO₂-HAP electrochemically on Ti-6Al-4V alloy substrate. Metal substrate is connected to working electrode of system. Platinum

rod and Ag/AgCl were used as counter electrode to complete the circuit and reference electrode to measure potentials between reference electrodes and working electrode respectively. The deposition was carried out with designed levels of parameters 30 min, keeping the temperature of electrolyte at 50 °C. After deposition, deionised water was used to wash the coated samples. Samples were dried in air after deposition. The experimental setup used is depicted in Fig. 1.



Fig. 1 Autolab Potentiostat, PGSTAT302N

E. Surface Microstructure Characterization

The microstructure of the coated samples were examined directly by scanning electron microscope JEOL JSM-IT 100 at 20KV with EDS facility to determine the element composition.

F. Micro Vickers Hardness

Vickers micro hardness test were performed on Mitutoyo Vickers Micro Hardness Tester to evaluate micro hardness of composite coating. Micro hardness Test was conducted on Vickers hardness tester by applying a load 0.2kgf load for duration of 15 seconds.

IV. RESULTS AND DISCUSSION

A. Assessment of Micro Vickers Hardness

Vickers micro hardness test were performed on Mitutoyo Vickers Micro Hardness Tester to evaluate micro hardness of composite coating. Micro hardness Test was conducted on Vickers hardness tester by applying a load 0.2kgf load for duration of 15 seconds. Three readings were taken and average of readings was considered for statistical analysis. Results of micro hardness as well as means and S/N ratios for composite coating of TiO₂-HAP on Ti-6Al-4V obtained after the experiment are shown in the Table IV.

TABLE IV MEAN MICRO HARDNESS AND S/N RATIOS FOR TiO₂-HAP COATED Ti-6Al-4V

S.No.	Input Parameters			Responses	
	Voltage(V)	pH of Electrolyte	TiO ₂ Concn. (g/L)	Mean Micro Hardness	S/N Ratios
1	1	3.3	3	417.60	52.423530
2	1	4.3	4	581.43	55.283520
3	1	5.3	5	664.17	56.443360
4	1.5	3.3	4	564.07	55.025580
5	1.5	4.3	5	824.15	58.318540
6	1.5	5.3	3	987.00	59.886340
7	2	3.3	5	431.07	52.689550
8	2	4.3	3	639.53	56.123600
9	2	5.3	4	710.50	57.037390

It is found that for TiO₂-HAP coated Ti-6Al-4V, maximum micro hardness obtained at input voltage of 1.5 V, pH of 5.3 and TiO₂ concentration of 3g/L (Experiment No. 6) and minimum micro hardness obtained at input voltage of 1 V, pH of 3.3 and TiO₂ concentration of 3g/L (Experiment No. 3).

B. 1) Means and S-N Ratios of Micro Hardness of Composite Coating

The averages of the output responses are compared for each case to deduce optimum level of operation. Signal-to-noise ratio method is used to consider variations due to factors. From the mean S/N response factor, optimized levels of parameters for higher output response can be evaluated and significant parameters were nominated according to assigned ranks depending on their impact on the output response. Average S-N ratios were calculated to obtain maximum micro hardness at optimized parameters levels, criterion of S/N higher-the better was selected and S/N ratio was calculated using equation (2):-

$$S/N = -10 \cdot \log \left(\frac{1}{n} \sum \frac{1}{y^2} \right) \quad \dots(2)$$

Where n represents the number of samples in a given experiment and y represents the response output of experiment. Main effects plot for means is shown in Fig. 2.

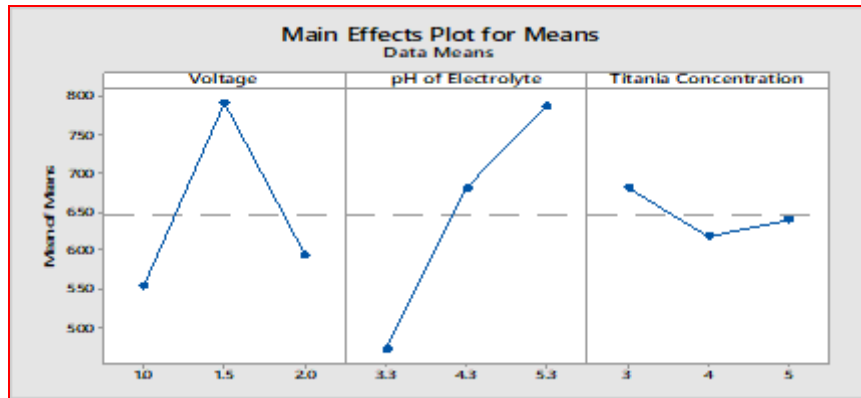


Fig. 2 Main effects plot of means for TiO₂-HAP composite coating on Ti-6Al-4V

It can be found that means are maximum at input voltage of 1.5 V, pH of 5.3 and TiO₂ concentration of 3g/L. Means are minimum at input voltage of 1V, pH of 3.3 and TiO₂ concentration of 5g/L. It has been observed that micro hardness increases with input voltage with almost constant slope from 1V to 1.5 V after that slope decreases up to 2V. Micro hardness was found to be maximum at input voltage of 1.5 V and decreases as voltage increases from 1.5 V to 2V. Micro hardness increases continuously with increase in pH value from 3.3 to 5.3. Micro hardness is maximum at titania concentration of 3g/L, decreases at titania concentration of 4g/L and a slight increase was observed at 5g/L. Main effects plot for S/N ratios are shown in Fig. 3.

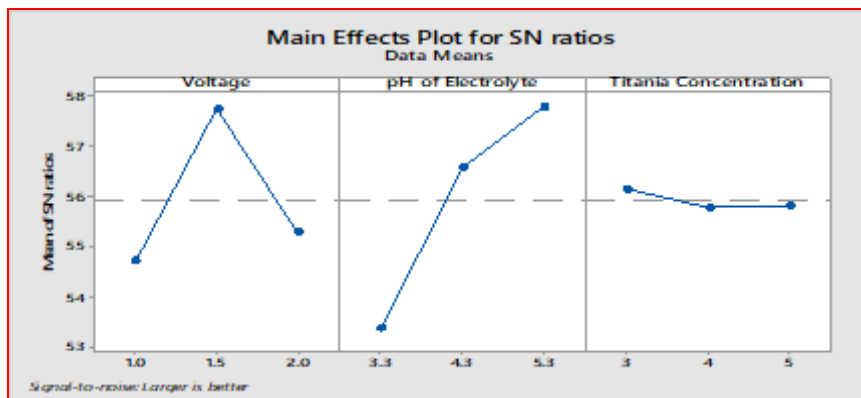


Fig. 3 Main effects plot of S/N ratios for TiO₂-HAP composite coating on Ti-6Al-4V

It can be noted that S/N ratio is maximum at input voltage of 1.5 V, pH of 5.3 and TiO₂ concentration of 3g/L. S/N ratio is minimum at input voltage of 1V, pH of 3.3 and TiO₂ concentration of 5g/L.

C. 2) Means and S/N Ratio Analysis of Micro Hardness of Composite Coating

The mean value of micro hardness for each level of process parameter is shown in the Table V.

TABLE V RESPONSE OF MEANS FOR TiO₂-HAP COATED Ti-6Al-4V

Response Variable	Level	Means		
		Voltage	pH of Electrolyte	Titania Concentration
Micro Hardness	1	554.3	471.0	681.7
	2	791.7	681.7	618.7
	3	594.0	787.3	639.7
	Delta (Δ)	237.3	316.3	63.0
	Rank	2	1	3

The difference among the highest mean and the lowest mean values of micro hardness for each level of parameters is designated as delta (Δ). Ranking of all factors are decided based on delta value in decreasing order. Rank 1 is given to factor with highest delta value and so on. The parameter with rank '1' was considered the most significant parameter. Similarly other significant parameters were decided. The ranks of the significant parameters are rated as pH of electrolyte (rank 1), voltage (rank 2) and titania concentration (rank 3). Relative parameter impacts on Means ratios depicts that pH concentration (Delta=316.3) has major impact on micro hardness while titania concentration has least impact (Delta=63.0). The mean value of S/N ratio response at each level of process parameter for micro hardness was calculated. Responses for S/N ratios for TiO₂-HAP coated Ti-6Al-4V are shown in Table VI.

TABLE VI S/N RATIOS RESPONSE FOR TiO₂-HAP COATED Ti-6Al-4V

Response Variable	Level	Mean S/N Ratio		
		Voltage	pH of Electrolyte	Titania Concentration
Micro Hardness	1	54.72	53.38	56.14
	2	57.74	56.58	55.78
	3	55.28	57.79	55.82
	Delta (Δ)	3.03	4.41	0.36
	Rank	2	1	3

Following the same procedure for calculation of delta (Δ) and rank as in case of means of micro hardness, the pH of electrolyte, voltage and titania concentration were ranked as 1, 2 and 3 respectively. pH of electrolyte solution was the most significant factor for optimum micro hardness. Relative parameter impacts on S/N ratios depicts that pH concentration (Delta=4.41) has major impact on micro hardness while titania concentration has least impact (Delta=0.36).

D. Analysis of Variance for Micro Hardness of Composite Coatings

In the present work, General Linear Model on MINITAB18 software has been used for the analysis. ANOVA of S/N ratios obtained for micro hardness test on TiO₂-HAP Coated Ti-6Al-4V samples showing percentage errors in the experiment is shown in Table VII.

TABLE VII ANOVA OF S/N RATIOS FOR TiO₂-HAP COATED Ti-6Al-4V

Source	DF	Adj. SS	Adj. MS	F-Value	P-Value	S	R-Sq	R-Sq (adj)
Voltage	2	96973	48486	32.93	0.029	38.3710	98.87%	95.50%
pH of Electrolyte	2	155613	77806	52.85	0.019			
Titania Concen.	2	6174	3087	2.10	0.323			
Error	2	2945	1472	-	-			
Total	8	261704	-	-	-			

The parameter with highest value of F ratio is the most significant parameter. It is found that for TiO₂-HAP Coated Ti-6Al-4V, pH is most dominating parameter (F=52.85) followed by input voltage (F=32.93) and Titania concentration (F=2.10) is least dominating parameter. Total value of sum of squares was found to be 261704 and out of this, sum of squares for errors was 2945 which is 1.12 % of the total sum of squares. So, for TiO₂-HAP Coated Ti-6Al-4, total error was found to be 1.12 % in the micro hardness test. The percentage contribution of titania concentration, voltage and pH of electrolyte were 0.323, 0.029 and 0.019 respectively. The model is significant as per the results are concerned.

E. Effects of Process Parameters on Micro Hardness

The process parameters considered for optimization for the process was voltage, pH of electrolyte and Titania concentration. 3 levels of each parameter were taken and electrodeposition was performed at these designed levels. The effect of these parameters on the properties of deposits has been analysed.

F. 1) Effect of Voltage on Micro Hardness

Voltage is the main process parameter that can affect the deposit properties. The effect of voltage on micro hardness of deposit is shown in Fig. 4.

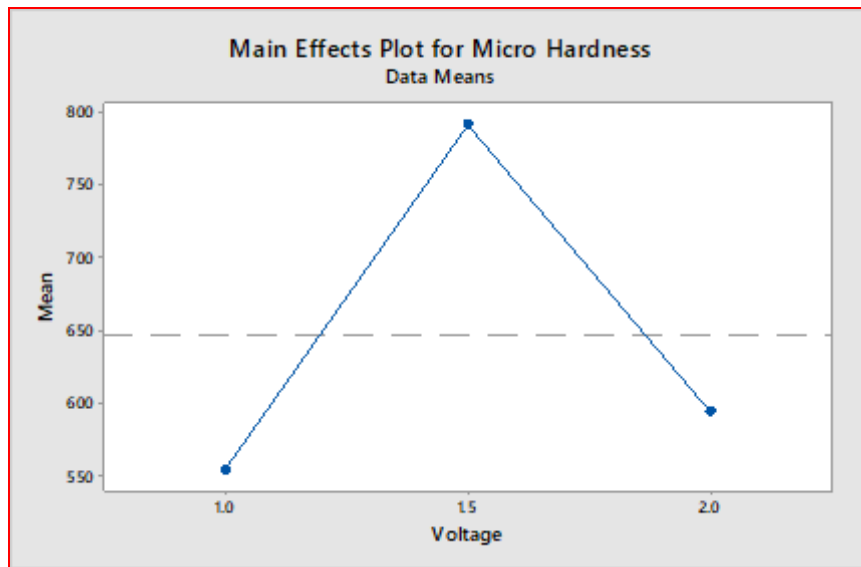


Fig. 4 Variation of micro hardness with voltage

Nucleation process accelerated on increasing the voltage and finer particles are obtained due to diffusion controlled growth process. During electro-codeposition, the TiO_2 particles are entrapped in between the space available in the calcium and phosphate ion matrix. Lower micro hardness is obtained at a voltage of 1V due low rate of deposition of TiO_2 , Ca and P ions. However higher micro hardness was obtained at 1.5V due to increase in rate of deposition of TiO_2 , Ca and P ions. Further increase in voltage has reduced the deposition rate and micro hardness of the coating. The micro hardness of the deposit was reduced at 2V due to erratic voltage magnitudes leading to lower deposition rate. The morphology changes from spherical/ globular particles of HAP with widely dispersed TiO_2 particles at 1V and flake type and needle like HAP crystals with entrapped spherical and flower like crystals of dispersed TiO_2 particles at 1.5 V and 2V. A dense hard and adherent microstructure with uniform dispersion of TiO_2 particles in HAP crystals were obtained..

G. 2) Effect of pH on Micro Hardness

pH is the main process parameter which affect the properties of electrodeposits. Figure 5 represents variation of micro hardness with change in pH value of electrolyte.

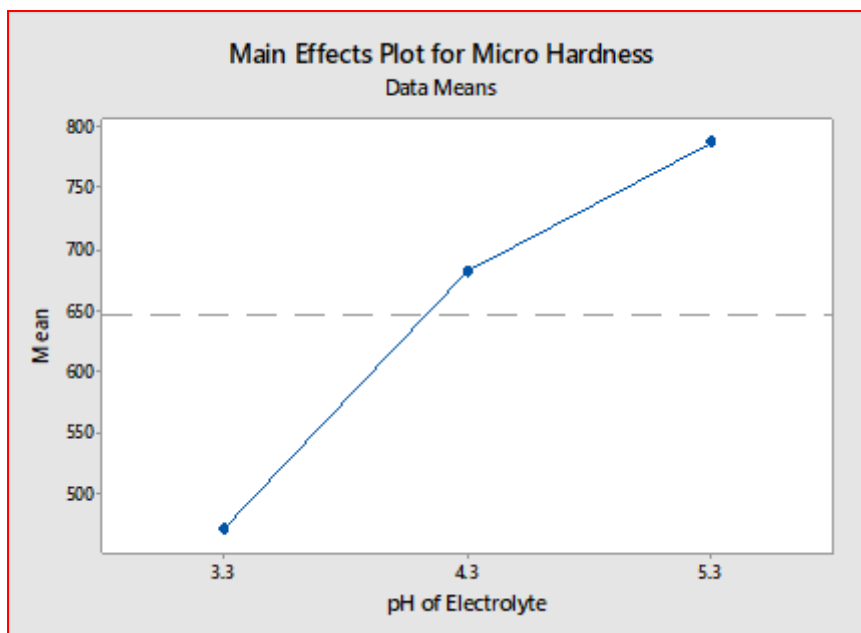


Fig. 5 Variation of micro hardness with ph of electrolyte

pH of the electrolyte has an adverse effect at lower levels content. The micro hardness of the deposit was increased as pH value increases from 3.3 to 5.3. The increased hardness may be due to increased co-deposition behaviour of TiO_2 , Ca and P particles at higher pH values. The greater micro hardness of the coating was achieved in pH 5.3 levels. These observations have described lower pH circumstances not suitable for composite plating with better micro hardness. At lower pH, hydrogen evolution took place, reduction process slowed down in the electrolyte and cathode efficiency of solution decreased. At higher pH values, evolution of hydrogen decreases thereby firm and hard deposits are obtained.

H. 3) Effect of Titania Concentration on Micro Hardness

The incorporation of TiO₂ nano particles in the Ca & P matrix leads to the formation of finer-grained structures, which further decreases the residual tensile stresses and increases the micro hardness and wear resistance of the composite coatings. Deposition was carried out by varying the concentration of TiO₂ nano particles in Watts' solution from 3 to 5 g/L at a designed voltage and pH levels. The micro hardness of each composite coating was measured and the maximum micro hardness was found at 3 g/L solution. The addition of more TiO₂ nano particles had a negligible effect on the coating hardness as shown in Fig. 6.

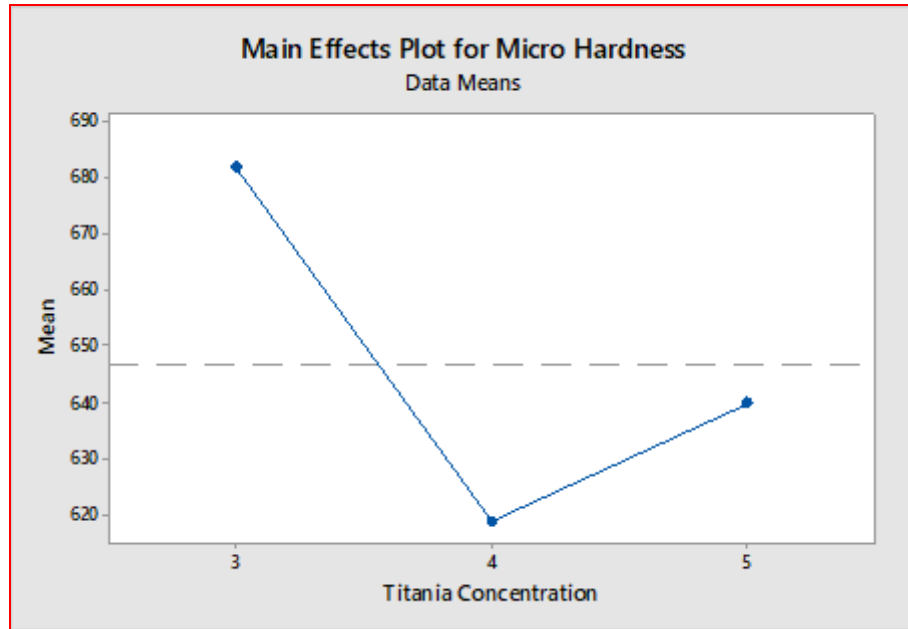


Fig. 6 Variation of micro hardness with titania concentration

When TiO₂ nano particles were added to the electrolytic bath of Watts' solution, the borders of the HAP grains became more localized, resulting in a reduction in mean grain size compared to pure HAP. The addition of TiO₂ nano particles to the HAP matrix changed the structure of the composite coatings. When higher concentration of titania particles are used, these particles remain in suspended form. The electrolyte solution becomes saturated at lower concentration of titania particles at 3g/L. The excess titania particles gets settle down in the electrolyte solution and does not participate in electrolysis process. Due to this reason, no major increase in micro hardness of composite coating was observed at higher titania concentration.

I. 4) Optimized Parameters for Electrodeposition

The effect of various parameters has been studied along with values of micro hardness obtained at various levels of these parameters. The optimized parameters values are shown in Table VIII.

TABLE VIII OPTIMUM PARAMETERS

S.No.	Parameters	Optimum Value	Micro Hardness			S/N Ratios		
			Optimum Value	Predicted Value	% Error	Optimum Value	Predicted Value	% Error
1	Voltage	1.5 V	987.00	967.33	1.99	59.88	59.85	0.05
2	pH of Electrolyte	5.3						
3	Titania Concentration	3 g/L						

J. Linear Regression Analysis

The responses are obtained at different values of process variables. Linear regression analysis of responses is done to achieve a linear fit between input parameters and output responses. The regression equation for micro hardness is represented by the following equation (3):-

$$Y = \beta_0 + \beta_1 a + \beta_2 b + \beta_3 c \quad \dots(3)$$

Where y is the response (micro hardness), β_0 is a constant, $\beta_1, \beta_2, \beta_3$ are coefficients. 'a', 'b' and 'c' represents absolute value of voltage, pH of electrolyte and titania concentration used during experimentation. The coefficients are given in Table IX.

TABLE IX COEFFICIENTS FOR RESPONSES

Response	β_0	β_1	β_2	β_3
Vickers Micro Hardness	-9	40	158.2	-21.0

Putting the values of all constants in the regression expression, final equation (4) can be written as:-

$$Y = -9 + 40a + 158.2b - 21.0c \quad \dots(4)$$

In regression equation, micro hardness of the coating depends mainly on pH of electrolyte followed by applied voltage and Titania concentration.

V. CONCLUSIONS

- (i) The Taguchi's approach L9 orthogonal array methodology was used to investigate the influence of parameters on micro hardness of electrodeposited TiO₂-HAP composite coatings on Ti-6Al-4V. The effect of the process parameters on the micro hardness was evaluated using Taguchi method. The pH of electrolyte solution was found to be the most significant parameter.
- (ii) The optimal combinations of the process parameters for maximum micro hardness were determined. The mean S/N ratio values are computed for process parameters and their levels. The delta values of factors are ranked by highest to lowest. It is recognized that the pH of electrolyte solution is the most significant factor for the response
- (iii) Micro hardness of uncoated Ti-6Al-4V which was 350 HV initially, found to be increased to a value of 987 HV in case of electrodeposited substrate.
- (iv) Significant increase in micro hardness of the Ti-6Al-4V was observed after electrodeposition, due to presence of TiO₂ particles. Using optimum parameters, micro hardness can be increased.
- (v) From S-N ratios analysis, the optimal setting of the influencing parameters for micro hardness is: voltage-1.5 V, pH of electrolyte-5.3, titania concentration-3g/L.
- (vi) There is 182% increase in micro hardness of Ti-6Al-4V after composite coating.
- (vii) Release of V ions from Ti-6Al-4V alloy into body was prevented due to TiO₂-HAP composite coating, which was a serious concern for application of the alloy requiring high service life.

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