Int. J. Electrochem. Sci., 17 **(2022)** Article Number: 220418, doi: 10.20964/2022.04.05

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

Multi Objective Optimization of Pulse Electrodeposition of nano $Ni\text{-}TiO_2$ and $Ni\text{-}Al_2O_3$ Coating on Inconel 617

D.R. Joshua[1,*], *A. Jegan*[2] *and A. Srinivasan*[3]

[1] Department of Mechanical Engineering, AVS Engineering College, Salem-636003, Tamil Nadu, India.
[2] Department of Mechanical Engineering, Sona College of Technology, Salem-636005, Tamil Nadu, India.
[3] Department of Mechanical Engineering, AVS Engineering College, Salem-636003, Tamil Nadu, India.
[*]E-mail: joshuamec@gmail.com

Received: 3 December 2021 / *Accepted:* 17 January 2022 / *Published:* 4 March 2022

Inconel 617 gets employed in aerospace industry and high temperature applications. Inconel's favorable properties provide it huge scope and usage is in various fields. For enhancement of properties, nano-coating is highly preferred on Inconel 617. Pulse electrodeposition method of nano coating is widely used to achieve such coatings. The plan of the present work was to optimize the deposition parameters in the multi objective way. $Ni\text{-}TiO_2$ and $Ni\text{-}Al_2O_3$ nano composite coatings on Inconel 617 were performed. For obtaining high micro hardness (MH) and good surface finish (Ra), TOPSIS multi objective technique was used to optimize the process parameters. Based on extensive study on past research work, duty cycle, current density and frequency were considered as inputs. L18 run order is used as experimental design to perform the work. Based on TOPSIS optimization, $Ni\text{-}TiO_2$ electrodeposition coating, Frequency of 50Hz , 40% Duty cycle and 0.2 A/Cm^2 current density were identified as optimal parameters for obtaining high hardness and better Ra. Hardness value of 474Hv and Ra of 0.412 µm were achieved in the confirmation experiment.

Keywords: Optimization, Inconel 617, Aluminium oxide, Titanium dioxide, Pulse Electrodeposition, MH and Ra.

1. INTRODUCTION

The scope of application of Inconel 617 keep extending to various fields. Usage of Inconel 617 in aerospace and petrochemical industries is increasing day by day [1]. The surface quality is one of the key factors that affect the functioning and durability of engineering materials and components.

Corrosion, wear and fatigue processes are known well to cause defects in most of the components. To improve the surface quality of any material, coating is the proven method all over the world [2]. Protective coating methods are classified as electroplating, thermal spray technique and vapor deposition technique etc. For enrichment of material properties such as hardness, wear and corrosion resistance in metallic and nonmetallic materials, electroplating coating technique is the effective way [3]. In material electroplating, co-deposited particle size is the dominant factor that impacts the coating properties. Minimum size particles provide enhanced coating properties. Nano size particle deposition on the metal improves the homogeneous deposition of the coating [4]. Many hard oxides such as SiO_2 (silicon dioxide), Al_2O_3 (Aluminium oxide) and TiO_2 (Titanium dioxide) etc. have attracted the researchers because of their proven capability for performance enhancing properties of materials. These oxides are used in material deposition to enrich hardness, wear and corrosion-resistance of the base material [5]. In material electrodeposition, current density, bath composition, frequency and duty cycle are the most influencing parameters [6]. Coating quality on the base material depends on controlling or varying of the process parameters. Better microstructural characteristics and particle distribution are achieved by appropriate variation in the process parameters [7]. Baghery [8] performed a research in Ni-TiO_2 nano coating and have stated that uniform distribution of TiO_2 nano particle was noticed in the coating which resulted in high corrosion resistance and wear performance. Isil Birlik [9] prepared Ni-TiO_2 nano composite and performed material characterization by electrodeposition technique. Based on experimental results of the study, better corrosion resistance was achieved with 0.3 A/Cm^2 current density. Hyun Cho [10] examined the influence of refractory ceramic coating on Inconel 617. Based on the experimental observations, Al_2O_3 was identified as promising coating option for Inconel 617 particularly in heat transport applications. Thermal corrosion resistance was also found to increase with Al_2O_3 coating. El-Awadi [11] conducted an experimental research on hot corrosion behavior of Inconel 617 and stated that major oxides get induced at high temperature.

Jegan [12] performed parametric optimization of electrodeposition of Ni/nano Al_2O_3. Based on the results, duty cycle was identified as the dominant factor with respect to material hardness. Taguchi approach was used to predict the influence of process parameters. The combination of 20Hz frequency, 30% of duty cycle and 0.4A/Cm^2 of current density was identified as optimum parameters. Natrajan [13] have developed a numerical model for identifying the characteristics of Ni-SiC coating on AISI 1022 and observed duty cycle being the dominant factor compared to current density and frequency. Past literature shows that many research works have been performed on Inconel 617 with the aim to improve corrosion resistance and wear. Nano Al_2O_3 and TiO_2 were used separately in electrodeposition coating for enrichment of both, wear and corrosion resistance of Inconel 617 [14-20]. Not many research works were found to focus on improving surface roughness and material hardness through deposition. The aim of this work is to enhance material hardness and reduce surface roughness by electroplating of nano Ni-Al_2O_3 and Ni-TiO_2 on Inconel 617. Parametric optimization is also planned to obtain best possible results through TOPSIS method of optimization. For achieving multi objective optimization, TOPSIS is a proven and prominent method [21-26].

2. MATERIALS AND METHODS

The Inconel 617 material composition is given in Table.1. Material Density of Inconel 617 is 8.36g/cm^3 and the Melting range is 1332-1380°C. Hardness of the selected Inconel 617 base material is 172 HRB and Tensile strength 831 MPa. The Yield strength and Modulus of elasticity are 410 MPa and 211 GPa respectively.

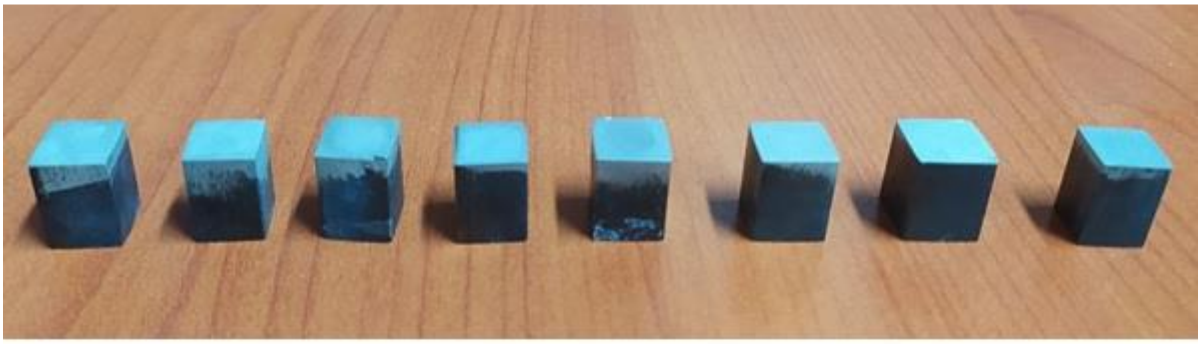
Table 1. Material Composition of Inconel 617

Inconel 617	**Ni**	**Cr**	**Co**	**Mo**	**Bal.**
Weight (%)	50.8	22.7	10.85	9.25	6.40

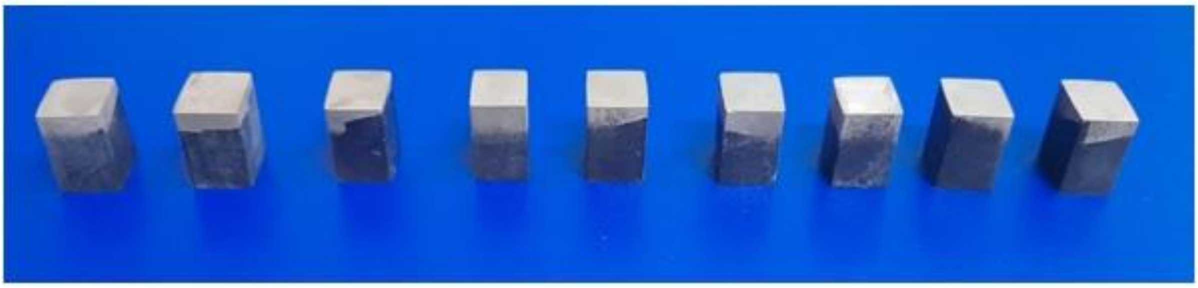
The Inconel 617 work-material was shaped to the dimension of 10 x 10 x 30 mm specimen using Wire cut EDM process. The specimen preparation of Inconel 617 material included polishing with the help of silicon carbide abrasive paper of grade size 80-2500 followed by ultrasonic cleaning for 15min with Acetone. Finally the top surface is again cleaned using distilled water in room temperature. In this experimental work, TiO_2 and Al_2O_3 particles were used with the average size of 100nm for electro deposition.Ni-TiO_2 and Ni-Al_2O_3 were electrodeposited on the specimen with Watts-type electrolyte for 10 microns of coating thickness. Table 2 indicates the bath composition used for experiments. The coated specimen is given in Fig.1.

Table 2. Bath Compositions of Experiments

S.No	**Electroplating bath composition**		**Value**
	Ni-TiO_2 Coating	Ni-Al_2O_3 Coating	
1	Nickel Sulphate ($NiSO_4.6H_2O$) (gl^{-1})		300
2	Nickel Choloride ($NiCl_2.6H_2O$) (gl^{-1})		50
3	Boric Acid (H_3BO_3) (gl^{-1})		30
4	Sodyumdodecyl sulfate (SDS) (gl^{-1})		0.1
5	TiO_2 nanoparticle ($d_m \leq$ 100nm) (gl^{-1})	Al_2O_3 nanoparticle ($d_m \leq$ 100nm) (gl^{-1})	10
6	Temperature (°C)		55
7	pH		4
8	Plating time (min)		11.5



a) Ni-TiO_2 Coated specimen



b) Ni- Al_2O_3 Coated specimen

Figure 1. a) Ni–TiO_2 Coated Specimen b) Ni-Al_2O_3 Coated Specimen

The experiments were performed utilizing dynatronix pulse generator having a power range of 110-120V AC single phase and with the frequency range of 50 to 60 Hz. This work mainly focused on obtaining optimized coating parameters to enrich the desired properties. The input parameters chosen were Frequency, Duty cycle and Current density. For producing accurate results, coating was also included as one of the parameter. The experiments were conducted as per design of experiments. Mitutoyo-SURFTEST SJ-410 roughness tester was used to measure the roughness value and hardness was measured with Vickers Hardness Tester. Micro hardness was measured with 10kg play load. The experimental process parameters used and the measured outputs are presented in Table 3. Table 4 respectively.

Table 3. Coating parameters and their levels

Symbol	Control factors	Level 1	Level 2	Level 3
A	Composite coatings	Ni-TiO_2	Ni- Al_2O_3	
F	Frequency (Hz)	30	40	50
D	Duty cycle (%)	30	40	50
I	Current density (A/Cm^2)	0.2	0.4	0.6

Table 4. Experimental Results

S. No	Composite coatings	Frequency (Hz)	Duty cycle (%)	Current density (A/Cm^2)	Micro hardness (Hv)	Surface Roughness (µm)
1	Ni-TiO_2	30	30	0.2	432	0.478

2	Ni-TiO_2	30	40	0.4	462.66	0.516
3	Ni-TiO_2	30	50	0.6	484.33	0.553
4	Ni-TiO_2	40	30	0.2	450	0.405
5	Ni-TiO_2	40	40	0.4	507.3	0.406
6	Ni-TiO_2	40	50	0.6	483	0.497
7	Ni-TiO_2	50	30	0.4	464	0.412
8	Ni-TiO_2	50	40	0.6	472	0.484
9	Ni-TiO_2	50	50	0.2	463.7	0.492
10	Ni- Al_2O_3	30	30	0.6	357	1.298
11	Ni- Al_2O_3	30	40	0.2	383.3	1.146
12	Ni- Al_2O_3	30	50	0.4	397.66	1.383
13	Ni- Al_2O_3	40	30	0.4	374.66	1.3956
14	Ni- Al_2O_3	40	40	0.6	406.6	0.803
15	Ni- Al_2O_3	40	50	0.2	389	0.938
16	Ni- Al_2O_3	50	30	0.6	400.33	0.823
17	Ni- Al_2O_3	50	40	0.2	374.3	0.948
18	Ni- Al_2O_3	50	50	0.4	411	0.883

3. RESULT AND DISCUSSION

3.1 Single parameter optimization

a) S/N Ratio

The objective of this work is to achieve high hardness and lesser surface roughness on the coating of Inconel 617. In single objective optimization, finding the signal to noise ratio (S/N ratio) plays a vital role in obtaining the influence of parameters.

Table 5. Response Table for SN Ratios-MH

Level	**Composite coatings**	**Frequency (Hz)**	**Duty cycle (%)**	**Current density (A/Cm^2)**
1	53.41	52.41	52.28	52.34
2	51.77	52.72	52.70	52.75
3	-	52.65	52.80	52.69
Delta	1.64	0.31	0.52	0.41
Rank	1	4	2	3

Minitab 19 is used to calculate the S/N ratio. For High hardness and lesser surface roughness, higher the better and smaller the better methods are used respectively. The appropriate expressions for finding S/N ratios are shown in equation 1 and 2 respectively.

Smaller the better-------------- $S/N = -10\log\frac{1}{n}\left(\sum_{i=1}^{n} y_i^2\right)$ (1)

Higher the better------------------ $\frac{S}{N} = -10\log\left(\sum\left(\frac{1}{Y^2}\right)/n\right)$ (2)

Where, "n" indicates number of experiments performed and "y" indicates the data. The calculated S/N ratio of MH is given table 5.

Based on the calculated S/N ratio, rank was given as per Delta value. From Table.5 it is noticed that, in obtaining high hardness, selection of composite coatings plays a major role. After composite coatings, other dominant factors that play significant role for achieving high hardness are Duty cycle followed by current density ranked in that order. Frequency range did not produce much impact on the material hardness. It is obvious that, hard Nano material deposition over the base material produces high hardness. At the same time varying duty cycle is the key factor to control the material deposition [13]. It was noted that, first level of composite coatings, second level of frequency, third level of duty cycle and second level of current density have high influence on the coating process. On the basis of response Table 5, nano Ni-TiO_2 composite coatings, 40 Hz frequency, 50% duty cycle and Current density of 0.4 A/Cm^2 were identified as optimal level parameters for achieving high hardness. Varying the duty cycle certainly change the amount of movement of ions. This movement variation affects the deposition rate on the specimen [17].

Table 6. Response Table for SN Ratios – Ra

Level	Composite coatings	Frequency (Hz)	Duty cycle (%)	Current density (A/Cm^2)
1	6.58	1.84	3.08	3.38
2	-0.38	3.55	3.51	2.77
3		3.90	2.70	3.14
Delta	6.96	2.06	0.81	0.61
Rank	1	2	3	4

Similarly, for obtaining parameter levels to achieve lesser surface roughness (Ra), S/N ratio was calculated and presented in Table 6. Based on S/N ratio, the order of domination is: Composite coatings > Frequency (Hz) > Duty cycle (%) > Current density (A/Cm^2). Current density does not have much impact on Ra. On the basis of response Table (Table 6), nano Ni-TiO_2 composite coatings, 50 Hz of frequency, 40% duty cycle and Current density of 0.2 A/Cm^2 were identified as optimal level parameters for achieving better surface finish. High current density affects the material deposition rate and also creates poor surface quality on the coated specimen. These results are in agreement with Jegan [12] results.

Table 7. Analysis of Variance

a) MH						
Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**	**PC (%)**
Composite coatings	1	29212.7	29212.7	176.81	0.000	82.49
Frequency (Hz)	2	782.0	391.0	2.37	0.144	2.21
Duty cycle (%)	2	2202.5	1101.3	6.67	0.014	6.22
Current density(A/Cm^2)	2	1562.7	781.4	4.73	0.036	4.41
Error	10	1652.2	165.2			4.67
Total	17	35412.1				100.00
b) Ra						
Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**	**PC (%)**
Composite coatings	1	1.60480	1.60480	60.12	0.000	76.85
Frequency (Hz)	2	0.15556	0.07778	2.91	0.101	7.45
Duty cycle (%)	2	0.02551	0.01276	0.48	0.634	1.22
Current density(A/Cm^2)	2	0.03545	0.01772	0.66	0.536	1.70
Error	10	0.26693	0.02669			12.78
Total	17	2.08824				100.00

Analysis of Variance (ANOVA) was performed to attain the parameters' contribution to hardness and surface-roughness and given in Table.7. From Table 7, it is noted that, composite coatings contribute significantly on Micro Hardness (MH) and Ra. Its contribution is comparatively higher compared to other parameters. The contribution of composite coatings to MH and Ra is 82.49 % and 76.85 % respectively. It can be inferred that, selection of appropriate coating material is very important in the improvement of MH and Ra. At the same time Duty cycle (6.22%) also significantly contributes towards high hardness and frequency contributes 7.45% on Ra. Homogeneous material deposition of nano particles on Inconel 617 is achieved by increasing the duty cycle. It produces high hardness on the base metal. Minimum frequency range implies the role of flow of ions and it directly reflects in the surface roughness. Ra was improved with maximizing the frequency range. This result is matching with that of Cheng [18].

3.2. Multi Parameters Optimization - TOPSIS analysis

TOPSIS method is the most common method of multi objective optimization. Initially, decision matrix is required to perform the TOPSIS study. It is expressed in rij. Expression used for decision matrix is given in equation 3. In next step, assigning weightage for each response is required. Followed by assigning weight, normalized value is found from decision matrix and response weightage. It is given in equation 4. Here $aij =$ th run order value 'j'. Here wi is noted as weightage of ji.

$$.r_{ij} = \frac{a_{ij}}{\sqrt{\sum_{i=1}^{m} a_{ij}{}^2}} \qquad (3)$$

$$Vij = Wi \, X \, rij \qquad (4)$$

In second step, S+ Positive ideal solution and S- negative ideal solution was found based on equation 5and 6.

$$S_i^+ = \sqrt{\sum_{j=1}^{M} (v_{ij} - v_j^+)^2} \quad (5)$$

$$S_i^- = \sqrt{\sum_{j=1}^{M} (v_{ij} - v_j^-)^2} \quad (6)$$

In final stage of TOPSIS, Closeness Coefficient (CC) value obtained indicates closeness coefficient. It is found with the help of equation 7 and rank is given based on higher CC value.

$$CC_i = \frac{S_i^-}{S_i^+ + S_i^-} \quad (7)$$

In this TOPSIS method, equal weightage is given to all factors for obtaining high hardness and lesser surface roughness. Calculated normalization value and CC of TOPSIS are given in table 8. Maximum value of CC is seen in the run order five with a CC value of 0.999. Run order 13 gives the minimum CC value of 0.0326. The run order based on CC values is: 5>7>6>4>8>9>2>3>1> 14>16>18>15>17>11>10>12>13. It is found that, high hardness and lesser surface roughness on Inconel 617 could be achieved by selecting appropriate level of factors such as composite coatings – Frequency (Hz)-Duty cycle (%)-Current density (A/Cm^2) which have dominance in this order. Compared to Ni-Al_2O_3, electrodeposited composite coatings of Ni-TiO_2 produced significant impact on MH and Ra. This is due to homogeneous deposition of Ni-TiO_2 on Inconel 617 compared to Ni-Al_2O_3. Nano Hard oxide deposition depends on movement of ions with respect to duty cycle [19-20].

Table 8. Normalized, Separation measures and CC values

Exp. no	Normalization		Weighted normalized		Separation measures		CC*
	MH	Ra	MH	Ra	S+	S-	
1	0.2364	0.1338	0.1182	0.0669	0.02299	0.13006	0.8498
2	0.2531	0.1444	0.1266	0.0722	0.01976	0.12646	0.8649
3	0.2650	0.1548	0.1325	0.0774	0.02165	0.12297	0.8503
4	0.2462	0.1134	0.1231	0.0567	0.01568	0.14097	0.8999
5	0.2776	0.1137	0.1388	0.0568	0.00014	0.14448	0.9990
6	0.2643	0.1391	0.1321	0.0696	0.01449	0.13041	0.9000
7	0.2539	0.1153	0.1269	0.0577	0.01189	0.14075	0.9221
8	0.2583	0.1355	0.1291	0.0677	0.01468	0.13141	0.8995
9	0.2537	0.1377	0.1269	0.0689	0.01705	0.12980	0.8839
10	0.1953	0.3634	0.0977	0.1817	0.13158	0.01366	0.0941
11	0.2097	0.3208	0.1049	0.1604	0.10912	0.03567	0.2463
12	0.2176	0.3871	0.1088	0.1936	0.14013	0.01126	0.0744

13	0.2050	0.3907	0.1025	0.1953	0.14332	0.00483	0.0326
14	0.2225	0.2248	0.1112	0.1124	0.06215	0.08405	0.5749
15	0.2128	0.2626	0.1064	0.1313	0.08132	0.06464	0.4429
16	0.2190	0.2304	0.1095	0.1152	0.06542	0.08102	0.5533
17	0.2048	0.2654	0.1024	0.1327	0.08426	0.06283	0.4271
18	0.2249	0.2472	0.1124	0.1236	0.07190	0.07325	0.5046

Table 9. Analysis of Variance-CC*

Source	DF	Adj SS	Adj MS	F-Value	P-Value	PC (%)
Composite coatings	1	1.45591	1.45591	69.65	0.000	78.63
Frequency (Hz)	2	0.12994	0.06497	3.11	0.089	7.02
Duty cycle (%)	2	0.03638	0.01819	0.87	0.448	1.96
Current density(A/Cm^2)		0.02023	0.01011	0.48	0.630	1.09
Error	12	0.20904	0.02090			11.29
Total	17	1.85149				100.00

Based on CC* Value, the combination $A_1F2D_2I_2$ is found as the optimum level of parameters for obtaining high hardness and better roughness. Results of ANOVA for CC* is given in Table 9. From ANOVA result, it is noticed that composite coatings dominate for 78.63% compared to other experimental parameters. Option of frequency decides the flow of ion and its contribution on material deposition is 7.02%. Duty cycle and current density contribute 1.96% and 1.09 % respectively. Apart from composite coatings, all other parameters exhibit similar level of impact on nano coating. These values are in agreement with Arunsunai Kumar [21].

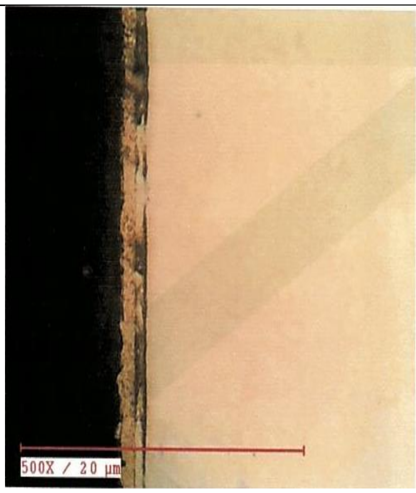
3.3. Confirmation Experiment

In every parameters optimization process, confirmation experiment is the final stage to predict the accuracy of optimized results. Table 10 presents the results of confirmation experiment. Based on the plotted results, $A_1F_2D_2I_2$ was found as the optimized levels. Ni-TiO_2 electrodeposition coating, Frequency of 50Hz, 40% Duty cycle and 0.2 A/Cm^2 Current density were identified as optimal parameter levels for obtaining high hardness and better Ra. Confirmation experiment was performed with optimum parameters and it produced the hardness value of 507.3Hv and Ra of 0.406 µm. Hardness value and Ra were improved by 17% and 15% respectively. For detailed surface study, SEM analysis was done (Scanning Electron Microscopic analysis) on the coated specimen done in optimum condition. Coated Inconel 617 and its SEM images are seen in Fig.2 and Fig.3 respectively. It is noted that, homogeneous material deposition was achieved and with defect free surface. XRD test was carried out at optimum conditions and it is given in Fig.4. It clearly shows the, fine texture of nano particles, defect free surface that produced maximum hardness in optimum condition [27].

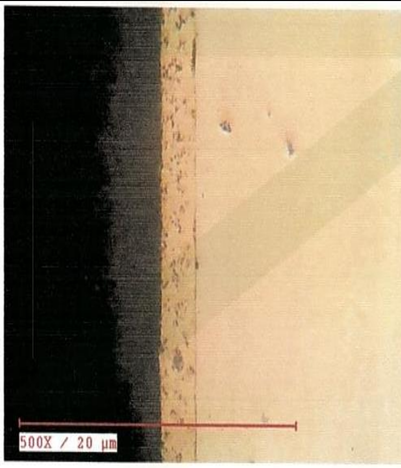
Table 10. Results of confirmation experiment

Parameters	Initial process parameters	Optimal process parameters	
		Prediction	Experiment
Levels	$A_1F_1D_1I_1$	$A_1F_2D_2I_2$	$A_1F_2D_2I_2$
MH	432	-	507.3
Ra	0.478	-	0.406
CC*	0.850	0.936	0.999
Improvement of CC*: 0.149			

a) Inconel 617



b) $Ni\text{-}TiO_2$ coating thickness



c) $Ni\text{-}Al_2O_3$ coating thickness

Figure 2. Coated Inconel 617 with coating thickness under optimal conditions

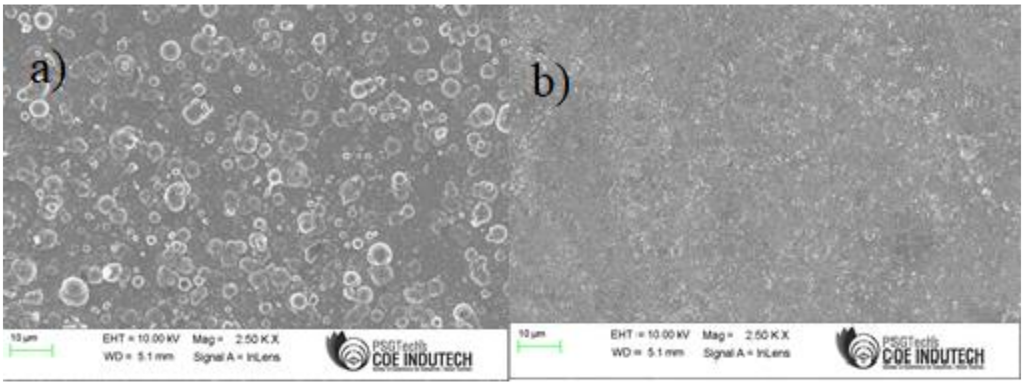
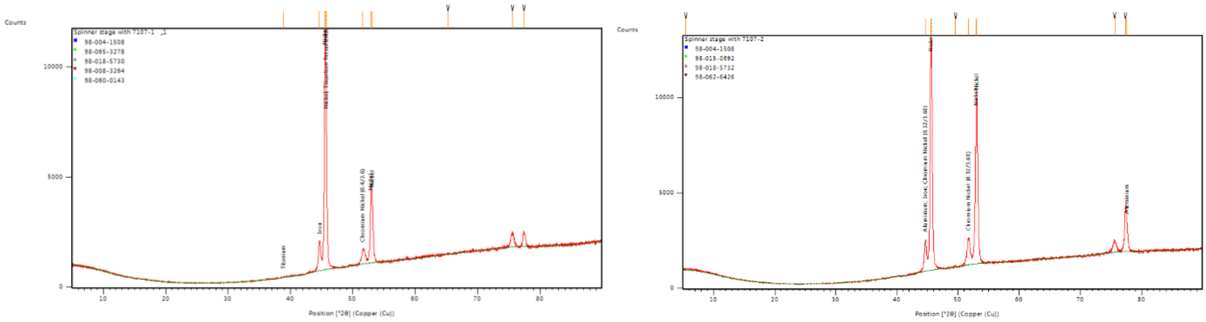


Figure 3. SEM Analysis (a) $Ni\text{-}TiO_2$ (b) Ni- Al_2O_3 with F=40Hz, D= 40% and I= 0.4 A/Cm^2.



(a) (b)

Figure 4. XRD Images – (a)Ni-TiO_2 (b)Ni- Al_2O_3 with F=40Hz, D= 40% and I= 0.4 A/Cm^2.

3.4 Corrosion Test

Corrosion test was done according to ASTM G28-02(RA15) standards [28-29]. The specimens were ground finished using 600 grit. The test Solution was prepared with 25gm of ferric sulphate [$Fe_2(SO_4)_3$] in which 400ml of distilled water and 236ml of sulphuric acid added. In the corrosion test, after 24 hours at 120^0 C and specimen was pickled with 20% HNO_3 + 5% HF solution at 60^0 C for 5 minutes before testing. The corrosion rate of Ni-TiO_2 coated specimen is 0.177 mm/month or 2.13mm/year whereas the corrosion rate of Ni-Al_2O_3 nano coated specimen is 0.237mm/month or 2.85mm/year. Ni-Tio_2 shows better resistance to corrosion compared to Ni-Al_2O_3. These test results are in best agreement with Kewther [30].

4. CONCLUSIONS

This work is aimed to find the appropriate nano electrodeposition coating on Inconel 617 to obtain maximum hardness and better Ra. From the outcomes of the experimental work, the conclusions arrived are given as follows.

1. For obtaining high hardness and minimum roughness from the coating on Inconel 617, Ni-TiO_2 composite coatings dominates over Ni-Al_2O_3 coating in providing better results.
2. Nano Ni-TiO_2 composite coatings, F =50 Hz, D = 40% and I = 0.2 A/Cm^2 were identified as optimal parameter levels for achieving better surface finish.
3. Ni-TiO_2 composite coatings, F = 40 Hz, D = 50% duty cycle and I=0.4 A/Cm^2 were found as optimal parameter levels for achieving high hardness.
4. TOPSIS study reveals that Ni-TiO_2 electrodeposition coating, F = 50Hz, D =40% and I = 0.2 A/Cm^2 were identified as optimal parameter levels for obtaining high hardness and better Ra.
5. In Optimum condition, Hardness value of 507.3Hv and Ra of 0.406 µm were achieved in the confirmation experiment. Hardness value and Ra were improved by 17% and 15% respectively.
6. The better surface quality and uniform material deposition of the nano-coating were ensured with SEM analysis and XRD technique.

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