

Effect studies on Electrodeposited Ni- Al₂O₃ composites by conventional and sediment type co-deposition methods using Taguchi approach

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Abstract— Ni-Al₂O₃ composite coatings were prepared from watts bath through conventional type and sediment type deposition. L9 orthogonal array experimental design was implemented for experimental design. The plating parameters, current density, temperature of bath and particle concentrations were considered with three levels for both conventional and sediment type depositions. Experiments were accomplished by the run orders of L9 orthogonal array design. The deposited coatings were examined under optical, SEM micro graphs, XRD and EDX investigations to confirm the particle depositions in matrix. Micro hardness of deposits was investigated in Vickers micro hardness tester with the pay load of 100 gram force. The composite coatings were influenced with greater micro hardness than pure Nickel plating. The direct effects of plating parameters on micro hardness were investigated with mean effect studies, signal to noise ratio analysis and analysis of variance (ANOVA). The significances of process parameters for conventional and sediment type depositions were ranked by order and verified by three types of analyses. For conventional type deposition, current and temperature were the most significance factors on micro hardness and temperature and bath concentration for sediment type deposition. It is revealed that sediment type deposition produced grater micro hardness, than conventional type deposition.

Index Terms— Electro deposition; composite coating; micro hardness; S/N ratio; ANOVA

I. INTRODUCTION

In a conventional electro co-deposition technique, the electrodes were situated vertically in the plating cell and in a sediment type electro co-deposition the electrodes are situated horizontally one over the other with adequate inter-electrode distance so that articles settle on the electrode surface as sediment on the cathode as the metal deposition progress [1].

Various investigators had reported the formation of electro co deposited composite coatings. Two co-deposition methods were explored by Guglielmi [2] for composite coating. He proposed two methodologies of successive dispersion process for the entrapment of reinforcement

particles in electro deposition for Ni-TiO₂ and Ni-SiC composites.

The first step is that the particles were first loosely (weakly) adsorbed on the surface of the cathode due to Vander- Walls Weak forces (creating a pathetic physical bond between the particles and cathode) which effects in higher concentration of loosely clutched particles originated on the plated specimen. In the second step, the loosely held particles are trapped in the deposit as an effect of strong adsorption behavior by the consequently applied electrical field (Coulomb force) which eternally deposits the particle to the cathode surface and consequently the particles were covered with depositing metal ion phases.

Celis and roos [3] had established a representation for incorporation of particle in a metal matrix. They found five steps for the occurrence of codeposition of particles in metal matrix during electrocodeposition process: i) codepositing particles are surrounded by ionic cloud patterns; ii) mass transfer of reinforcement particles to the hydrodynamic boundary layer by convection mode; iii) Due to the diffusion incidence, mass transfer of particles to the cathode location; iv) Due to the magnetism of free ions and electrodynamic ions adsorbed on the particles in the cathode side, and v) electro-diminution of adsorbed ions coupled with absorption of particles into the growing metal matrix. yahia and adel [4] illustrated that the incorporation of reinforcement particle in the metal matrix due to; i) transformation of bulk amount of particles from electrolytic solution to cathode surface, ii) adsorption behavior of particles with the electrically charged cathode, iii) dispersion of particles in the continuous growing metal matrix layer. Along with, Vereecken [5] developed a model for deposition of particle in metal matrix by electrocodeposition process. Fransaer [6] formulated an analytical model for deposition of particles in metal matrix through electrocodeposition approach. The proposed models were provided the basics of electro composites formations.

Aruna [10] had examined the deposition activities of in-situ impregnated alumina particles in Ni matrix, proposed that utilization of porous particles may be a better direction to improve the adhesion property of particles and micro hardness in the improvement of electrodeposited metal matrix composites. The properties of composite coatings primarily depend upon the matrix phases and also the amount and distribution of co-deposited particles within the matrix. Adequate level of strengthened particles within the metal matrix directs enhancement of properties like micro hardness, wear resistance, tribological behaviors and corrosion resistance of the coatings. Jan Steinbach and Hans Ferkel [11] had organized the Ni-Al₂O₃ composite coating by DC and

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pulsed DC electroplating, decided that PDC technique had produced efficient smaller particle deposition. Saha and Khan [12] had conveyed that, weight fraction of Al₂O₃ increase with higher amplification of current density conditions, micro hardness of coating also increased with addition of particles inclusions in matrix. García el [13] had directed a comparative study on effects of mechanical and ultrasound agitation, detected that ultrasound agitation method absolutely affected the characteristics of electrodeposited Ni-Al₂O₃ composite coatings, when compared to composite coatings obtained from mechanical agitation. Particle concentration, current levels, effects of surfactant and pH were considered in Ni- Nano Al₂O₃ composite preparation by Gul [14], reported that were resistance and micro hardness of coating were partial by percentage of distribution of particles in matrix and process parameter conditions.

Haifeng Liu [7] had prepared Ni-Al composite coating with high Al content by sediment type co deposition. They concluded that 30-35% Al particles were incorporated in a composite coating. Particle loading in bath was the significant factor for the volume fraction of Al particles during the deposition. Suman and saho [8] had prepared the electro less Ni-B composite coating and examined the influences of process parameters on micro hardness of coating with the help of Taguchi analysis. They took four parameters; temperature of bath, reducing agent concentration, nickel concentration and annealing temperature, and design into an L27 orthogonal array to conclude the substances of process parameters on hardness of the coating with the help of analysis of variance, which depiction that annealing temperature and concentration of reducing agent have significant influence on hardness characteristics of electro less Ni-B coating.

Narasimman [9] had investigated that electro co-deposition process primarily affected by the parameters like current density, amount of ceramic particles within the plating bath, pH scale of the solution, temperature of bath and stirring speed. Many materials, ceramics; Al₂O₃, MoS₂, SiC, Si₃N₄ and TiO₂ [17-21]; metal powders; Al, Cr, Cu, Mo, Ti, V and W and cermet's [12-15]; hard diamond particles [23]; cost effective pumice particles for oxidation resistance [23]; PTFE for self lubrication [17]; have been deposited in various metal and alloy matrices.

Adabi and Amadeh [25] had investigated the electrodeposition mechanism of Ni-Al composites using Guglielmi's model revealed that zeta potentials of Al particle are similar to that of Al₂O₃ particles. Inclusion of Al particles alters the reduction potentials of Ni to additional negative values. The dislocation of in decrease potential is endorsed to a decrease in active surface area. The Ni-Al codeposition behavior obeys the Guglielmi's model.

Cheng yu [26] investigated the nucleation behavior of nickel deposition while codepositing the SiC particles. They concluded that nucleation time of Ni-SiC deposition may progressively be abridged with over potential intensification and be littler than that of pure nickel depositions. They also found inert particles have an influence on deposition behaviors and can refine the microstructure morphology of Ni-SiC composites.

The literature survey has explored the preparations and investigations of electro composites using electro-codeposition methodology. The deposition behaviors of various inert particles codeposited in several metal and alloy matrices have been studied from literature studies. However, previous experimental studies in composite coatings were accomplished by randomized manner. No robust experimental design was followed. Only few process parameters were considered in effect studies to identify the influences on micro hardness, volume fractions and coating thickness. Selection of process parameters also has not been done in proper categorization. In this study robust experimental design has been implemented for experimental design and significance studies. Three principal process parameters of electro deposition such as current density, bath temperature, and Al₂O₃ concentration in the bath with three levels were considered in the experiment to investigate the effects on micro hardness of Ni-Al₂O₃ composite coatings. The Taguchi method of L9 orthogonal array has been employed to categorize the influences of process parameters on micro hardness of Ni-Al₂O₃ coating using design of experiments approach. With these, Analysis of Variance (ANOVA) techniques has been applied to determine and confirm the significances of these parameters.

II. TAGUCHI APPROACH

Taguchi method involves reducing the variation in a process through robust design of experiment. The whole objective of the process is yield high quality product at low cost of industrialist. Taguchi established a technique for designing experiments to inspect in what way different parameters distress the mean and variance of a process performance characteristic that defines however well the method is functioning. The experimental proposal proposed by Taguchi comprises using orthogonal arrays to consolidate the parameters disturbing the process and the levels at which they should be contrasts. Instead of having to test all conceivable groupings like the factorial design, Taguchi approach tests of combinations. This agrees for the assemblage of the required data to regulate which factors most disturb product quality with a minimum amount of investigation, thus saving time and assets. The Taguchi method is best used when there are an intermediate number of variables (3 to 50), few exchanges between process parameters, and once solely a couple of variables impart suggestively.

The arrays are chosen by the number of parameters (variables) and its levels (states). Analysis of variance on the collected data from the Taguchi design of experiments can be used to select new parameter values to optimize the performance characteristic. By knowing the number of parameters and levels, the appropriate orthogonal array can be designated. In this work three plating parameters with three levels were taken for experimental work. Based on the above parameters and levels, L9 orthogonal array of Taguchi approach was implemented for robust experimental design. The objective of the present work is to maximize the micro hardness of the Ni- Al₂O₃ composite coatings. Since the larger is better module is personalized. The higher is well characteristic of S/N ratio can be formulated as:

$$\frac{S}{N} = -10 \log \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{y_{ij}^2} \right)$$

(1) Where n is equal to replication of the investigational work and y represents the output of experiment. In addition to mean effects techniques and ANOVA can be implemented to determine the effect of the process parameters on the performance characteristic.

III. MATERIALS AND METHODS

Schematic diagram of electroplating setup is shown in figure 1. Electro deposition experiments were carried in 2000 ml Borosil glass container. A watts type nickel plating bath employed for electrodeposition work. Mild steel plate of sized 75 x 25.4 x 1 mm³ thick was employed as a cathode substrate and area of deposition was taken as 25.4 x 25.4 mm² (1 inch²) rest behind portions were shrouded. A pure nickel plate was used as anode. The mild steel cathode plate was degreased by acetone and polished with dry cloth buffing wheel, for amputation of rust layer. Fine particles of Al₂O₃ (6-8µm avg.) composite powder with required quantity was assorted in nickel solution. The plating solution along with Al₂O₃ composite powder was agitated for 3 hours before plating for getting of homogeneous blend along with surfactant to ensure the codeposition. Each mild steel cathode plate was etched in cathodic and anodic cleaning bath for removal surface contamination in plating area and to confirm better adhesion of coating, and finally rinsed with distilled water, and kept immersed in plating bath [16].

The reinforcing elements were retained in suspension via mechanical agitation using a motorized stirrer. Speed of agitation was examined by use of digital tachometer and attuned by speed controller unit. A regulated D.C power supply machine (made by Royal Instruments, India, capacity: 0-30V and 0-2A) was engaged for electro deposition. pH of electrolyte was adjusted with pH meter (Made by Hanna, Mauritius) and adjusted to required level earlier the initiation of each plating. The pH value of the bath was attuned by use of diluted acidic or else base solutions. A hot plate with temperature controller unit (made by Royal Instruments, India, Capacity: AC type, 230 volt, 50 Hz, Temperature range: 30° to 110°C) was involved to heating up of bath to required temperature levels. A 'K'-type thermocouple was employed to observe the temperature of the bath during plating. The distance between Ni anode and mild steel cathode was reserved constantly. The time duration for each of plating was taken as 60 min. for all cases. pH of the bath for each plating was taken as 4 constantly. For conventional plating, electrodes were vertically positioned and sediment type positioned horizontally. The principle of electro co-deposition is identical to the fundamentals of electroplating. During plating Ni ions from the anode captured the inert particles in the solution and co deposit the same in cathode substrate and the composite coating was formed. The experiments were performed based on the run orders of L9 orthogonal array pattern and nine samples were prepared from bath for each type of deposition.

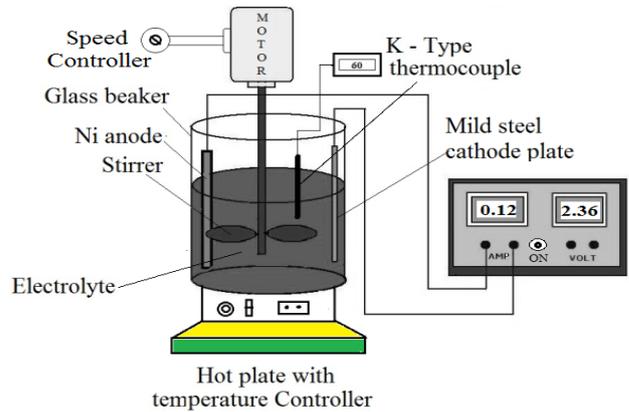


Fig.1 Electrodeposition setup

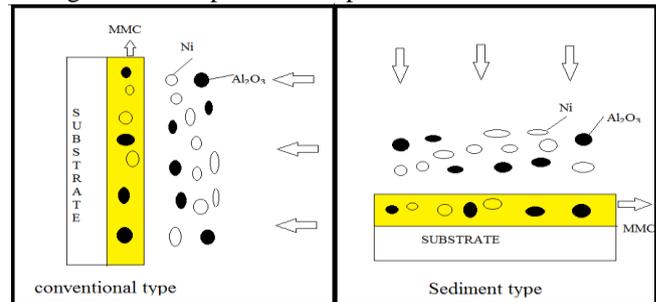


Fig.2 Conventional and Sediment type Depositions

Table I: parameters and levels

Parameters	Units	Levels		
		I	II	III
A. Current density	A/dm ²	1	2	4
B. Temperature of bath	°C	30	45	60
C. Bath concentration	g/l	15	30	45

IV. RESULTS AND DISCUSSIONS

A. Assessment of surface morphology, volume fraction of Al₂O₃ of Ni-Al₂O₃ composite coating

The coated samples were polished and cleaned by metallographic procedures for the investigation of surface morphological studies. The pattern of deposition of Ni-Al₂O₃ particles in the deposit were examined by scanning electron microscope (JEOL-Field emission SEM, model TSM-6701F, Japan) at different magnification. Volume percentages [16] of embedded alumina particles were determined from optical micrographs using high transmission metallurgical microscope (KYOWA, JAPAN, model:ME-LVX2, magnification range: 50-1000x)

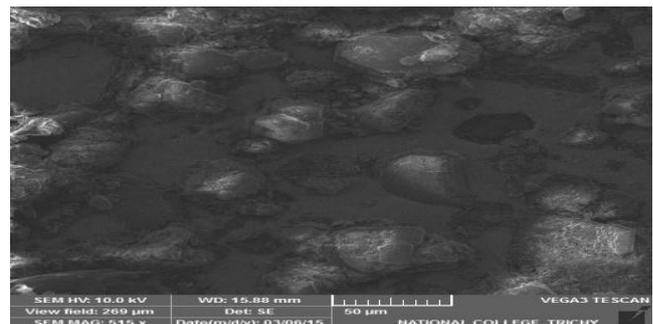


Fig.3 Micro structure for conventional type attained at current: 4 A dm⁻², temperature: 45°C, bath concentration: 15g L⁻¹ of Al₂O₃.

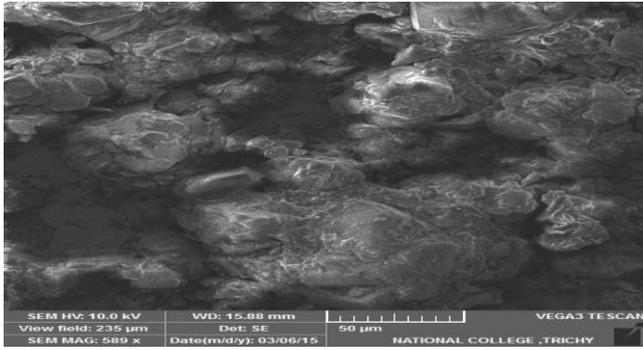


Fig.4 Micro structure for sediment type attained at current: 4 A dm⁻², temperature: 45°C bath concentration: 15g L⁻¹ of Al₂O₃.

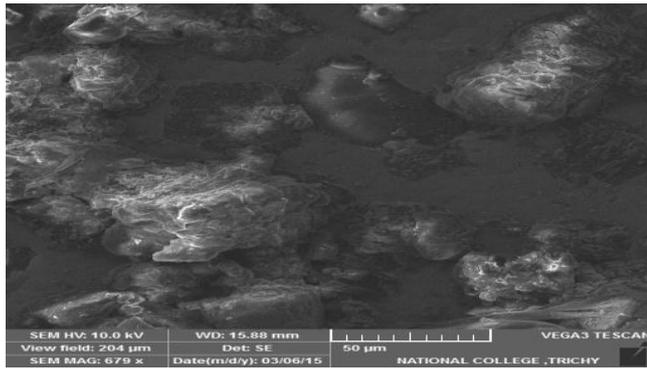


Fig.5 Micro structure for sediment type attained at current: 4 A dm⁻², temperature: 30°C, bath concentration: 45g L⁻¹ of Al₂O₃.

From above analysis, it was found that the particles were uniformly dispersed in nickel matrix with uniform grains. Fig 3 shows the uniform dispersion of Al₂O₃ particles in nickel matrix prepared from conventional type deposition. Fig 4&5 shows the uniform deposition of Al₂O₃ particles in nickel matrix prepared from sediment type deposition. The above investigations authorize the deposition of nickel and ceramic particle phases in the metal matrix composites.

The XRD and EDAX observations were accomplished to analyze the compositional phases in the coated samples. Fig 6 shows the XRD patterns of composite coating. It was conformed that presences of nickel and Al₂O₃ phases in the deposited layer by XRD patterns. Fig 7 shows the EDAX results of composite coating and confirms the presence of nickel and other compositional elements in the matrix.

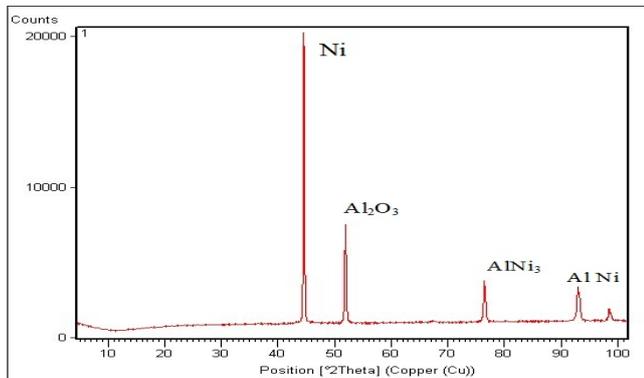


Fig.6 XRD Results of Ni-Al₂O₃

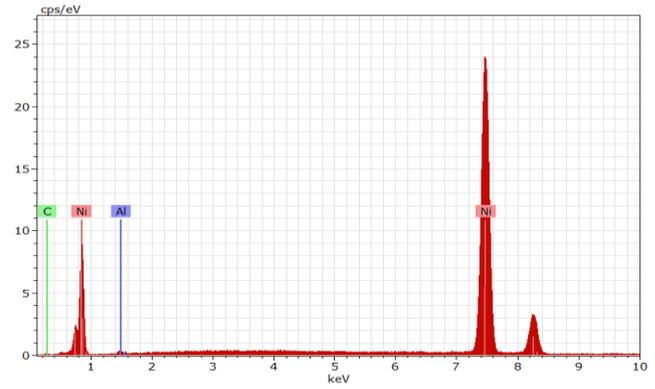


Fig.7 EDX Results of Ni-Al₂O₃

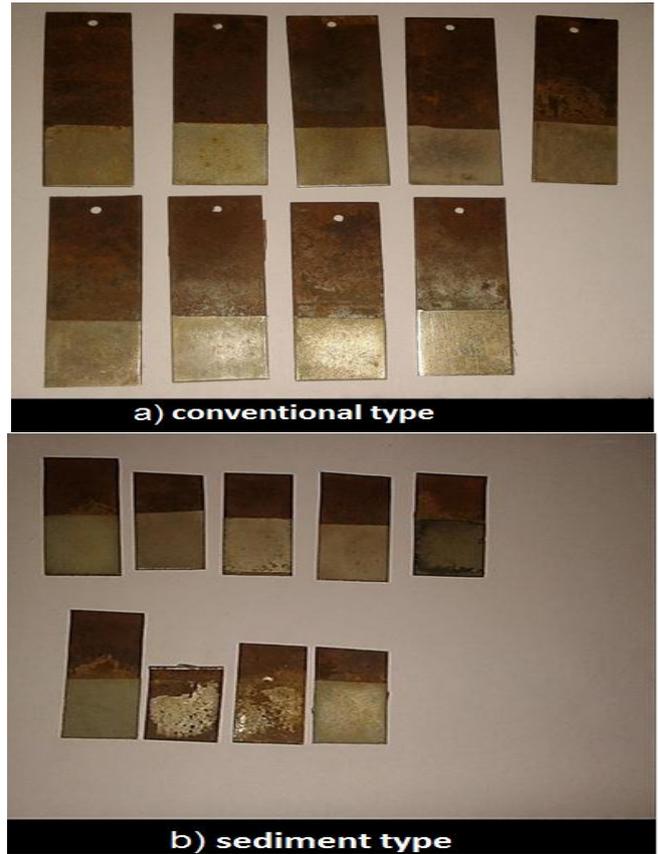


Fig.8 Samples prepared from a) conventional and b) sediment type depositions

B. Assessment of micro hardness of Ni –Al₂O₃ composite coating

Micro hardness of the coated samples were examined in Vickers micro hardness tester (model & maker: SHIMADZU -TYPE HMV- 1/-2, SHIMADZU Corporation, Japan) with the payload of 100 gram force for 10 sec. of indentation period. The indented location was focused with 400X Magnification and the slider positions were adjusted to the diagonal lengths of indentation. Finally, the micro hardness was calculated by a system based on and value was taken from digital read out. Micro hardness of each sample was examined with three trials and the average value was taken for ending documentation.

C. Analysis of S/N ratio

To determine the effect each variable has on the output, the signal-to-noise ratio, or the SN number, needs to be calculated for each experiment conducted. From the mean S/N response factor, the most promising plating conditions for each design parameters can be recognized and the process parameters can be ranked according to their influence on the response parameter. In this tentative design, micro hardness

of the deposit is the response variable which needs to be maximized and hereafter larger the better features was preferred for these experimental studies. After manipulation the S/N ratio for experiment trails, the average S/N ratio value was calculated for each factor and level using equation 1. The equation no. 1 can be employed for determination of S/N ratio with larger is better taguchi's analyze design.

Table II: Experimental responses of conventional type deposition

ExptNo	Current A dm ⁻²	Temperature °C	Bath Concentration g L ⁻¹	Mass of Deposition mg	Coating Thickness micron	Volume fraction of Al ₂ O ₃ (%)	Micro Hardness HV	S/N ratio for hardness dB
1	1	30	15	70.6	12.3	31.48	284	49.066
2	1	45	30	98	17.1	28.19	219	46.809
3	1	60	45	87.4	15.22	34.62	264	48.432
4	2	30	30	105.8	18.43	21.14	426	52.588
5	2	45	45	136.5	23.77	40.49	1018	60.155
6	2	60	15	135.4	23.58	29.86	213	46.568
7	4	30	45	250.2	43.58	33.07	482	53.661
8	4	45	15	225.3	39.24	48.4	1670	64.454
9	4	60	30	237.3	41.33	37.62	700	56.902

Table III: Experimental responses of sediment type deposition

Expt No	Current A dm ⁻²	Temperature °C	Bath Concentration g L ⁻¹	Mass of Deposition Mg	Coating Thickness micron	Volume fraction of Al ₂ O ₃ (%)	Micro Hardness HV	S/N ratio for hardness dB
1	1	30	15	53.6	9.33	29.86	356	51.029
2	1	45	30	84.5	14.72	50.83	2389	67.564
3	1	60	45	57.7	10.05	34.62	246	47.819
4	2	30	30	102.4	17.84	47.15	942	59.481
5	2	45	45	159.5	27.78	39.07	293	49.337
6	2	60	15	111.1	19.35	44.57	382	51.641
7	4	30	45	101.2	13.45	48.4	659	56.378
8	4	45	15	161.3	28.09	53.16	2541	68.1
9	4	60	30	304.1	52.96	45.87	242	47.676

Table IV: Mean S/N ratio values of parameters on micro hardness by conventional deposition

Level	A	B	C
1	48.10	51.77	52.36
2	53.10	57.14	52.90
3	58.34	50.63	54.08
DELTA	10.24	6.51	1.98
RANK	1	2	3

Table VI: Mean S/N ratio values of parameters on micro hardness by sediment deposition

Level	A	B	C
1	55.47	55.63	56.92
2	53.49	61.67	58.24
3	57.38	49.05	51.18
DELTA	3.90	6.51	7.06
RANK	3	1	2

Table V: Mean effects of parameters on micro hardness by conventional type deposition

Level	A	B	C
1	255.7	397.3	722.3
2	552.3	969.0	448.3
3	950.7	392.3	588.0
DELTA	695.0	576.7	274.0
% of effect of parameters	44.7 %	37.3 %	17.7 %
RANK	1	2	3

Table VII: Mean effects of parameters on micro hardness by sediment type deposition

Level	A	B	C
1	997.0	652.3	1093.0
2	539.0	1741.0	1191.0
3	1147.3	290.0	399.3
DELTA	608.3	1451.0	791.7
% of effect of parameters	21.33 %	50.89 %	27.77 %
RANK	3	1	2

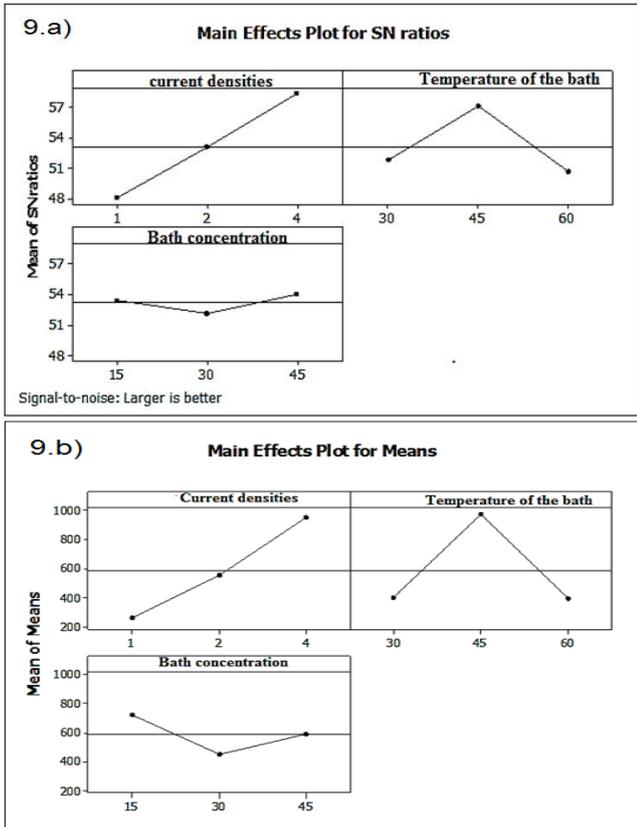


Fig.9 a) S/N ratio and b) mean effects plots for conventional type deposition

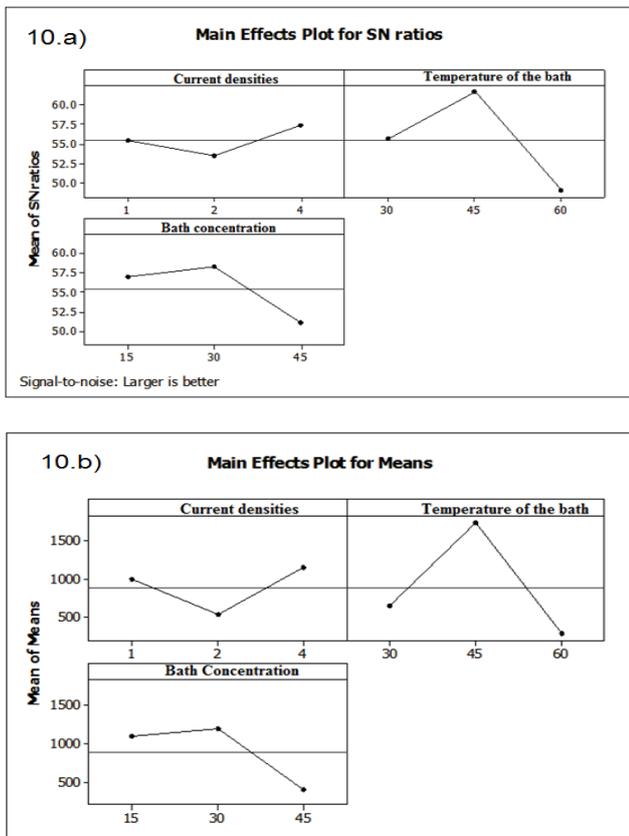


Fig.10 a) S/N ratio and b) mean effects plots for sediment type deposition

In this study MINITAB 16 software was employed for determination of S/N ratio and means of mean effects in design of experiments module. The effects plots were generated for S/N ratio and mean effects. The table-II shows

the experiment outcomes and S/N ratio values for micro hardness by conventional method and table-III shows the details for sediment type deposition.

Table-IV indicates the mean S/N ratio values and table -V indicates the means of response variable (micro hardness) for each level of each control factor for conventional type deposition. Similarly table VI&VII show the mean S/N ratio values and means for sediment type. The table V and VII indicated with percentage of effects of parameters on response. The mean response table for micro hardness for each level of process parameters was established in the united manner. Based on the mean value of the micro hardness for each level, the transformation between the maximum and minimum values was calculated. The maximum variance will give the most significant parameters, and rank for the significant parameters is illustrated. Mean effect plots for process parameters are shown in Fig.9 and Fig.10.

The significances of S/N ratio and mean effects response were shown in tables IV to VII. This table also comprises delta (Δ) which is the difference among the highest S/N ratio and the lowest S/N ratio values. Ranks for factors are allocated on the basis of the delta value. The highest delta value is assigned to rank 1; rank 2 is assigned to next highest delta value and the rest. Based on ranking positions, it was observed that current densities has the highest delta value, ranked by 1st position and identified as the most influencing factor on micro hardness in the conventional and temperature of the bath as the most influencing factor on micro hardness in the sediment type electro deposition. The means response tables (V&VII) indicate the percentage of effects of parameters on response (micro hardness), and the rank orders obtained for conventional and sediment type depositions were identical to the rank orders of S/N ratio analysis. In order to validate the results of S/N ratio and mean effects studies, ANOVA approach was implemented to identify the significances of process parameters on response variable.

D. Analysis of variance (ANOVA):

$$SS_T = \sum_{i=1}^n (\eta_i - \bar{\eta})^2 \quad (2)$$

Where 'n' is the number of experiment in the orthogonal array, ' η_i ' is the S/N ratio of the ith experiment and ' $\bar{\eta}$ ' is the total mean S/N ratio.

The percentage contribution of variance (ρ) can be calculated as follows;

$$\rho = (SS_D / SS_T) \quad (3)$$

Where SS_D is known as sum of the squares of deviation. F-test is a numerical tool (the mean square error to residual) in ANOVA used to found out the most important parameters that guidance the quality characteristic. Greater the F-value will be most powerful on the response quality characteristic P-value demonstrate the significance level (significant or non-significant) of the process parameter. In this study the ANOVA investigations for experimental response were

performed in ANOVA tool offered in MINITAB 16 software and the end results were tabulated.

Table VIII: ANOVA table for micro hardness conventional type deposition

source	DF	SS	F	p	ρ %	Rank
A	2	729706	1.93	0.341	38.8	1
B	2	659372	1.74	0.364	35.1	2
C	2	112628	0.30	0.770	5.99	3
		Error	2	378092		
		Total	8	1879798		

Table IX: ANOVA table for micro hardness - sediment type deposition

Source	DF	SS	F	P	ρ %	Rank
A	2	602434	0.37	0.729	8.9	3
B	2	3421882	2.11	0.321	50.6	1
C	2	1117514	0.69	0.592	16.52	2
		Error	2	1621130		
		Total	8	6762958		

Table-VIII shows the contribution levels (ρ %) of parameters on micro hardness analyzed from ANOVA module for conventional type deposition and table-IX for sediment type deposition. It is observed that, the contributions of parameters on micro hardness in conventional type deposition was about current density, ($\rho=38.8\%$); temperature of the bath, ($\rho=35.1\%$); bath

concentration of the Al_2O_3 , ($\rho=5.99\%$). For sediment type, temperature of the bath, ($\rho=50.6\%$); bath concentration of the

Al_2O_3 , ($\rho=16.52\%$); current density, ($\rho=8.9\%$). The above rank orders are good agreed with the rank orders of S-N ratio and mean effect studies. Thus, the influences and significances of process parameters on micro hardness of coating verified with three different schemes and validated.

V. CONCLUSIONS

Electrodeposited Nickel- Al_2O_3 composites have been produced from watts bath using conventional and sediment type electro depositions. Robust experimental design methodology has been implemented for effect studies for above depositions using Taguchi approach with the objective of less experimental trails and cost effective experimentation. Greater micro hardness has been attained in Ni- Al_2O_3 deposits than pure nickel coating. The following conclusions were established from the experimental and analytical studies.

- Micron sized Al_2O_3 particles were successfully dispersed in nickel matrix by conventional and sediment type electro depositions.
- L9 orthogonal array of taguchi's approach was engaged to frame the experimental trails with least number of experiments. The experiments were accompanied by changing the process parameters and levels established on the run orders.
- The surface morphological studies for Ni- Al_2O_3 coating was accomplished using optical micrographs and SEM

analysis in order to authorize the particle deposition in Ni matrix. With these, XRD and EDAX investigations were achieved to in order to approve presence of Al_2O_3 in nickel matrix.

- Experimental conclusions such as mass of deposit, coating thickness, volume fraction of particles and micro hardness of coating were studied systematically.
- The influences of process parameters on the response micro hardness were inspected via analytical studies such as mean effect studies and S/N ratio analysis. In order to authorize the end results of exceeding analysis, significance analysis studies were accompanied using statistical tool named ANOVA. The effects of process parameters were ranked by order.
- From the above examinations, current density and temperature of the bath are the most significance factor for the hardness for conventional type deposition; Temperature of the bath and bath concentration are the most significant factor for the hardness for sediment type depositions.
- It is essential to note that the mass of deposit was accomplished from 70.6 to 250.2 mg for conventional; 53.6 to 304.1mg for sediment type deposition. Volume fraction of particle found to be 21.14 to 40.49% for conventional; 29.86 to 53.16% for sediment type depositions. The micro hardness of Ni- Al_2O_3 coating was obtained in the span of 213 to 1248 HV from conventional deposition; 242 to 2541Hv from sediment type deposition.
- Such scientific and statistical approaches confirm the effects and significances of process parameters on end properties of coating or experimental responses. Thus improves the reliability of experimental investigations.

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