

Effects of Process parameters on Microhardness of Electrodeposited Ni-Fly ash-SiC Composite Coatings using Taguchi approach

S.Jeyaraj, V.Sudhambarish, Vallioor Sankaran Karthik, K.P.Arulshri

Abstract— Electrodeposited composite coatings also called functionally grade materials, have found many efficient applications in Engineering field due to their elevated micro hardness, corrosion and wear resistance. The present research work attempt has been made to codeposit fly ash particles with SiC in nickel matrix via electro deposition process. The influences of coating process parameters on microhardness of Ni- Fly ash + SiC are investigated with Taguchi approach. Four plating parameters, current density, pH, temperature of bath and particle concentration in bath are considered, and L_{27} orthogonal array is framed for experimental trials. The significances of process parameters and the direct effects on microhardness are investigated with the help of signal to noise ratio and analysis of variance, and ranked by order. The compositional studies on fly ash, surface morphological studies and phase structure studies are investigated with the help of scanning electron microscope (SEM), XRD and EDX analysis respectively. The obtained coating possesses higher microhardness in comparison with a pure nickel coating.

Index Terms— Electrodeposition, Composite coating, Microhardness, Taguchi method, Orthogonal array.

I. INTRODUCTION

Electrodeposition process is well known for their fabrication in micro and nano crystalline composites. This technique is increasingly used for many application and cost effective method for the manufacture of metallic and composite coatings [1]. This process has the advantages of low cost, low working temperatures, easy maintenance, ability to produce composite coatings and able to acquire different combinations of properties by just changing the process conditions [2],[19]. Several parameters can influence the electrodeposition process and consequently the microstructure of composite coatings, these parameters include electrolysis condition (composition of electrolyte bath, presence of pH, temperature, additives and electrolyte agitation), current conditions (DC or pulsed current, current density levels) and the properties of reinforcing particle (size, Engineering properties, concentration, type of distribution in bath) [3]. The metal matrix composites (MMC) are the materials in which the properties of the metallic host materials are modified with a addition of second phase material that can be a metallic or non

metallic powder, encapsulated particles or fibers [1],[4]. The MMC can be prepared by numerous methods like metal infiltration, powder metallurgy, hot pressing, diffusion bonding, spray deposition, stir casting, electro/electroless plating. However the major problem in the production of these materials is to obtain the wetting of reinforcement by the liquid metal which is very poor and is favored by a strong chemical bond at the interface. Electrodeposited MMC are also produced by the codeposition of inert particles like ceramics, polymers and fibers into a metal matrix from a electrolytic or a electroless bath [5].

The second phase particles can be hard oxides (Al_2O_3 , TiO_2 and SiO_2) or carbide particles such as (SiC or WC) [4]. Cheng yu *et al* [20] 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 had an influence on deposition behaviors and can refine the microstructure morphology of Ni-SiC composites. QiuyuanFeng *et al* [6] had investigated the influence of metal matrix phases and the amount of distribution of codeposited particles on the composite coating properties. They also suggested that the composite coating contents which can be improved by the addition of metal cationic accelerant and organic surfactant in an electrolytic bath and also it can be improved by changing applied current. However excessive use of surfactant leads to deposit brittleness and reduce in cathode area. The composite coating posses enhanced properties such as wear, corrosion, oxidation resistance, dispersion hardening or self lubrication, so that they can protect the metal substrates from severe environments during the applications [7], [8]. Susan *et al* [9] found that the smooth void free deposit can be produced when the codeposit particle size is small. Saha and khan [10] investigated the electro deposition of Ni- Al_2O_3 composites which revealed that the higher concentration and more uniform dispersion of particles in the metal matrix composites would improve mechanical, anti-corrosional, tribological and oxidation resistance properties of these composite coatings. The durability of coating material depends on wear resistance of the coating and also on the adhesion to the substrate, so it is important to evaluate the adhesive strength of the coating. The use of nickel for electroplating is becoming very common as large grained Ni is expected to deform easier whereas electrodeposited fine-grained structured nickel will resist [11]. Electrodeposited nickel has good mechanical properties such as high yield strength, excellent corrosion resistance, high electrical conductivity, good thermal conductivity, good magnetic property and hardness [5], [11].

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Temam *et al* developed a composite plating and identified that SiC particles can be codeposited with nickel to improve the mechanical and the corrosion behavior of steel substrates [12]. The codeposition of SiC particles into the Ni matrix leads to a considerable strengthening of nickel silicon carbide electro composites which prepared under optimum conditions with respect to pure nickel electrodeposit [13]. Substantial improvement in the mechanical properties such as roughness, hardness, scratch resistance and wear resistance could be obtained by using nano SiC particles in a nickel matrix [2]. The corrosion tests using the lost weight method show that the corrosion rate decreases with increasing SiC concentration whereas the microhardness increases with a higher concentration of silicon carbide particles in the nickel matrix [12]. In the electrodeposition process as, the codeposition of SiC nanoparticles increased as current density increases but decreased as current density passes through maximum value [13]. Current density and temperature have apparent effect on the grain size of nano crystalline nickel [14]. In this study, an attempt has been made for preparation of Ni-composite plating with SiC and fly ash (FA) particles suspended in watts bath. FA has been identified as an additive for improving mechanical and chemical properties of metallic alloys. Inclusion of FA in metals and alloys results in high abrasion, wear resistance and low friction coefficient [15].

The wear characteristics of zinc-fly ash coating were investigated by Panagopoulos *et al* using a pin-on-disk apparatus. The composite coating revealed increased wear resistance, than that of the pure zinc coating and the mild steel substrate. They investigated the adhesion strength between the zinc-fly ash coating and the mild steel substrate using scratch testing apparatus. It was found that adhesion strength between zinc-fly ash composite coating and the mild steel substrate higher compared to pure zinc coating to mild steel [16]. Fly ash is dependent on the concentration of the deposit in which it is deposited. Due to the deposition, the mechanical and electrochemical properties of the coating were increased [15], [17]. Ni-Co-fly ash composite coatings were by Panagopoulos *et al*, composed with crystalline Ni-Co solid solution and dispersed fly ash particles over a 5083 aluminium alloy substrate. The co-deposition of fly ash particles shows a momentous increase of the microhardness of the coating. Also, chemical analysis of the Ni-Co matrix confirmed that it composed of 80 wt. % Ni and 20 wt. % Co. The corrosion studies of the Ni-Co-fly ash/zincate coated aluminium alloy, in a 0.3 M NaCl solution (pH = 3.5), was investigated with potentiodynamic corrosion experiments [18].

In this study micro sized fly ash and SiC particles were deposited in nickel metal matrix using electro codeposition technique over a mild steel substrate. The influences of process parameters on microhardness of coating were investigated with signal to noise ratio analysis and means effects studies and the parameters were ranked by order. In order to confirm the ranking positions, analysis of variances (ANOVA) test was performed, resulted the similar rank orders of S-N ratio and means effects and confirms the significances of parameters.

II. TAGUCHI METHOD

Dr. Taguchi has formulated a method based on orthogonal array experiments which gives much reduced variance for the

experiment with optimum settings of control parameters through robust design of experiments. The general intention of the method is to produce high quality product at low cost to the manufacturer. Taguchi created means for experimental design to investigate how different parameters affect the mean and variance of a process performance characteristic that defines how efficiently a process is functioning [19]. This experimental design proposed by Taguchi which involves the use of orthogonal arrays to organize the parameters affecting the process and the levels at which they should be changed. As an alternative to test all possible combinations like the factorial design, the Taguchi method analyses pairs of combinations. This permits for the collection of the essential data to determine which factors affect more severely the product quality with a minimum amount of experimentation, thus saving time and experimental resources. The Taguchi approach is best suited when there are an intermediate number of variables (3 to 50), few interactions among variables, and when there are only a few variables that contribute significantly. Thus the combination of design of experiments with optimization of control parameters to obtain best results is achieved in the taguchi Method. Orthogonal Arrays (OA) provide a set of well balanced (minimum) experiments and taguchi's Signal-to-Noise ratios (S/N), which are log functions of preferred output, give out as objective functions for optimization, which helps in data analysis and forecasting of optimum results. Knowing the number of parameters and the number of levels, the appropriate orthogonal array can be chosen. In this investigation, four plating parameters with three levels were considered for experimental study. In this study, based on the parameters and levels given in table I, L27 orthogonal array of Taguchi approach was chosen for robust experimental design. The objective of the present work is to maximize the micro hardness of the Ni- SiC+Flyash composite coatings. Since the larger is better module was tailored. The higher is better characteristic of S/N ratio can be formulated as:

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

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

III. METHODS AND MATERIALS

Electro deposition experiments were carried in 2000 ml MERC glass container. The plating electrolyte was watts type nickel bath. Mild steel plate of sized 50.8 x 25.4 x 1.8 mm³ thick was employed as a cathode substrate and area of deposition was taken as 25.4 x 25.4 mm² and rests behind portions were masked. 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. SiC and flyash micro meter sized (7 to 9µm) was prepared homogeneously in a 1:1 ratio by ball milling process for a period of 8 hours. FA is an offshoot of thermal electric plants. The compositions of fly ash was investigated with XRD analyser and given in Fig. 5(b) and it was found that FA

composed with several ceramic phases. The required quantity of the composite powder was mixed in the Ni solution. The plating solution along with SiC + flyash composite powder was agitated for 3 hours before plating for getting of harmonized blend along with brightener and additive to ensure the co deposition. 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. Fig.1 shows the schematic of experimental setup.

The reinforcing particles were kept in suspension via mechanical agitation using a motorized stirrer. Speed of agitation was monitored by use of digital tachometer and attuned to 250 rpm constantly using speed controller unit. A regulated D.C power supply machine (made by Royal Instruments, India, capacity: 0-30V and 0-2A) was employed for electro deposition. pH of electrolyte was attuned with pH meter (Made by Hanna, Mauritius) and adjusted to required level before the commencement of each plating. The pH value of the bath was adjusted 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 engaged 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 retained constantly. The time duration for each of plating was taken as 60 minutes for all cases. For conventional plating, electrodes were vertically positioned. The experiments were performed based on the run orders of L27 orthogonal array pattern. The principle of electro co-deposition is identical to the fundamentals of electroplating. During plating ions from the anode was carried by the solution and deposited on the cathode as a thin film which acts as the protective coating. The experimental data set and the outcomes were given in table II. The formation of electrodeposited composite coatings given in Fig. 2.

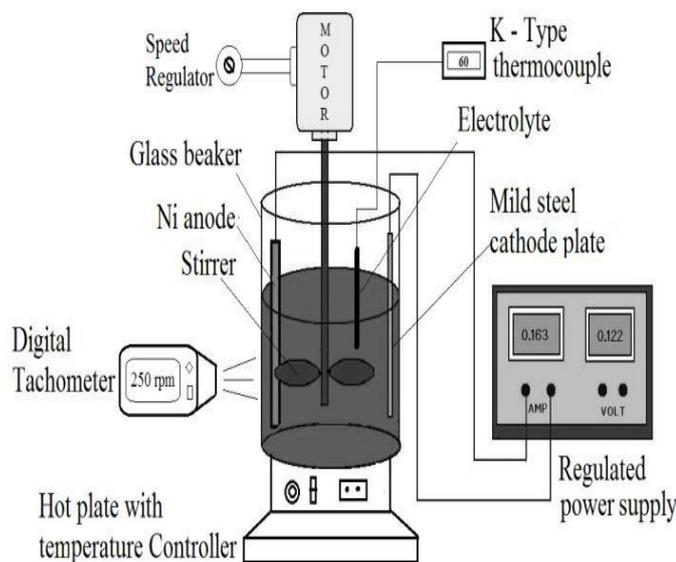


Fig. 1: Electroplating setup

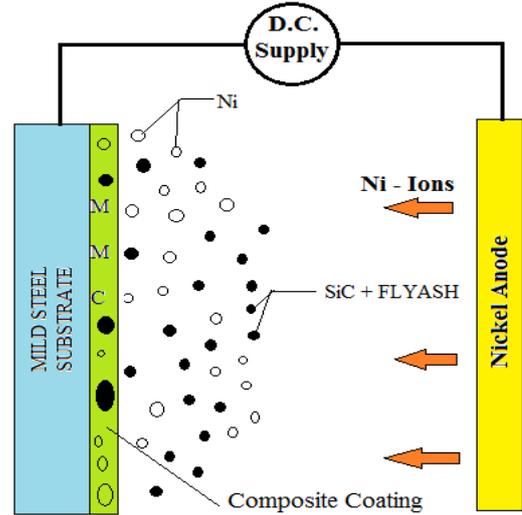


Fig. 2: Formation of Electrodeposited composite coating

IV. ASSESSMENT OF SURFACE MORPHOLOGY

The surface morphological examinations of the coated samples were investigated via metallographic procedures. The distribution of the particles (SiC & Flysh) in the Ni matrix were examined with the help of scanning electron microscope with various magnifications. It was confirmed that the particles were uniformly dispersed in nickel matrix. Fig.3 and 4 show the SEM micro graphs of composite plating, observed that particles were uniformly embedded in nickel matrix.

Table I: Parameters and Levels

Parameters	Units	Levels		
		I	II	III
a. Current density	A/dm ²	1	3	4
b. pH of bath	pH	2.5	3.5	4.5
c. Temperature of bath	°C	30	45	60
d. Bath concentration	g/l	10	20	30

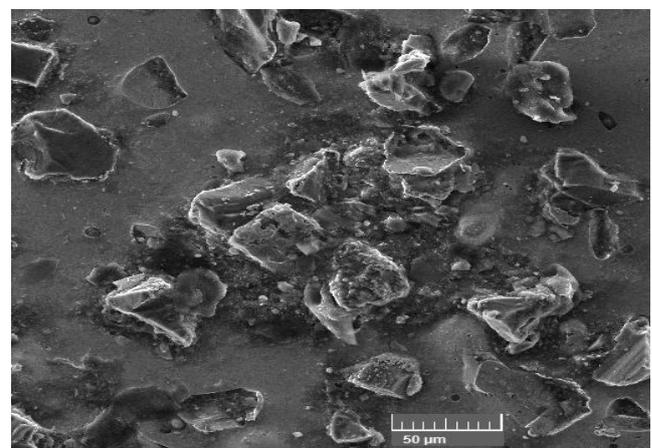


Fig. 3: SEM micrograph attained at current: 4 A dm⁻², pH: 4.5, temperature: 60°C, bath concentration: 30 g L⁻¹ of SiC+ Flyash

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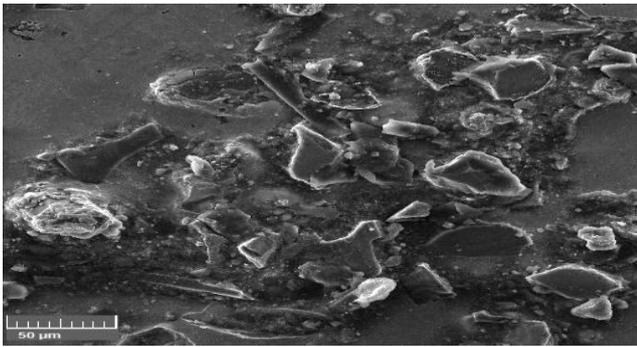


Fig. 4. SEM micrograph attained at current: 4 A dm⁻², pH: 4.5, temperature: 60°C, bath concentration: 30 g L⁻¹ of SiC+ Flyash

In order to confirm the presence of nickel and SiC +FA particles, EDX test were conducted. The samples were cut in to 10mm² area and investigated under EDX analysis. In general, EDX observations are made to investigate the compositional phases in the deposition. Fig.5.a) shows the EDX results of composite coating and confirms the presence of nickel and SiC and fly ash compositional elements in the matrix.

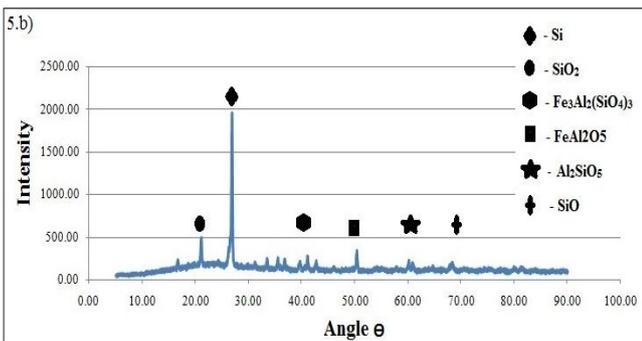
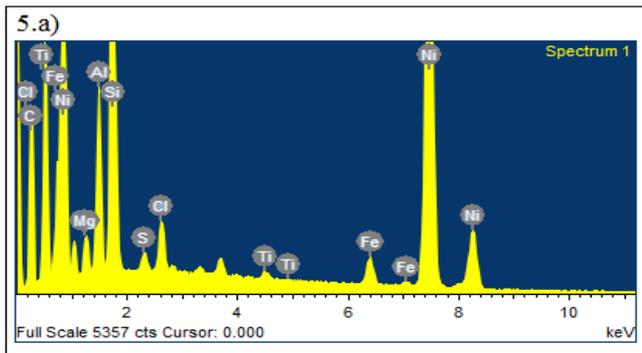


Fig. 5: a) EDAX Results of Ni-SiC+Flyash; b) XRD patterns of Fly ash

V. ASSESSMENT OF MICRO HARDNESS

The samples after thorough polishing were checked for indentations and then the hardness test was carried out. Micro hardness of the coated samples were examined in Vickers micro hardness tester (model & maker: SHIMADZU - TYPE HMV- 1/-2, Japan) with the load of 100 gram force for 10 seconds of indentation period. The indented location was focused at 400X. Based upon the diagonal lengths of the indentation the magnification and the slider positions were

adjusted. The microhardness was calculated and the value was taken from the digital read out. Micro hardness of each sample was inspected with three trials at different locations and the average microhardness value taken for documentations.

Table II: Experimental data and response

Expt. No.	Control Parameters					Mass of Deposit (mg)	Micro Hardnes (HV)	S/N ratio (dB)	Mean
	a	b	c	d	e				
1	1	1	1	1	1	55.9	367	51.28	366.5
2	1	1	1	1	2	53.2	217	46.70	216.5
3	1	1	1	1	3	88.6	216	46.66	215.5
4	1	2	2	2	1	57.6	223	46.94	222.5
5	1	2	2	2	2	68.3	224	46.98	223.5
6	1	2	2	2	3	72.4	254	48.07	253.5
7	1	3	3	3	1	53.2	215	46.62	214.5
8	1	3	3	3	2	64.3	297	49.44	296.5
9	1	3	3	3	3	63.8	235	47.42	235
10	2	1	2	3	1	186.5	467	53.37	466.5
11	2	1	2	3	2	192.2	469	53.42	469
12	2	1	2	3	3	162.3	292	49.30	292
13	2	2	3	1	1	173.5	320	50.10	320
14	2	2	3	1	2	314.8	258	48.23	258
15	2	2	3	1	3	190	657	56.34	656.5
16	2	3	1	2	1	177.7	518	54.28	518
17	2	3	1	2	2	217	519	54.30	519
18	2	3	1	2	3	161.8	528	54.44	527.5
19	3	1	3	2	1	266	107	60.59	1071.
20	3	1	3	2	2	178.3	635	56.05	635
21	3	1	3	2	3	157.1	318	50.03	317.5
22	3	2	1	3	1	245.4	463	53.30	462.5
23	3	2	1	3	2	244.3	594	55.47	594
24	3	2	1	3	3	248.2	721	57.15	721
25	3	3	2	1	1	241.3	450	53.06	450
26	3	3	2	1	2	271.1	824	58.31	824
27	3	3	2	1	3	230.4	889	58.97	889

VI. ANALYSIS OF S/N RATIO

It is essential to investigate the S/N ratio factor [6] from the experimental statistics to compute the average S/N ratio response for each experimental factor. The mean S/N ratio helps us to determine the apt plating conditions for each design parameters and thereby ranking the process parameters according to their impact on the response parameter. In this experimental design, to maximize the micro hardness of deposit of SiC+Flysh which is the response variable larger the better characteristics was preferred for this experimental investigations. Minitab 16 software was employed for taguchi analysis. After manipulation of S/N ratio for experiment trails, the average S/N ratio value was calculated for each

factor and level using equation 1. The experiment outcomes and S/N ratio values for micro hardness are given in Table II. Table III gives the average S/N ratio of micro hardness and rank orders of parameters based on the delta values. 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 density has the highest delta value, ranked by 1st position and identified as the most influencing factor on micro hardness of Ni-SiC + fly ash coating. The effects plot for S/N ratio given in Fig.6.

Table III: Mean S/N ratio of Micro hardness

Level	Current density	pH	Temp.	Bath Cont.
1	47.80	51.94	52.18	54.32
2	52.65	51.40	52.10	51.93
3	55.89	52.99	52.05	50.08
Delta	8.09	1.58	0.13	4.25
Rank	1	3	4	2

The mean response for micro hardness for each level of process parameters was developed in the integrated manner. Based on the mean value of the micro hardness for each level, the difference between the maximum and minimum values was calculated. The percentage of effects also calculated for process parameters. The maximum difference will give the most significant parameters, and rank for the significant parameters is depicted.

Table IV: Means of microhardness

Level	Current density	pH	Temp.	Bath Cont.
1	249.3	450.0	454.7	575.0
2	447.4	412.4	448.4	441.9
3	662.7	497.1	456.4	342.6
Delta	413.4	84.7	8.0	232.4
% of effect	55.98	11.47	1.08	31.47
Rank	1	3	4	2

Table IV indicates rank orders based on percentage of effects and delta values for mean of the response variable (micro hardness) for each level of control factor. Mean effect plot for process parameters is specified in Fig.7. For hardness based on ranking positions, it was observed that current density of the bath has the highest delta value, ranked by 1st position and identified as the most influencing factor on micro hardness of coating and others were ranked by order.

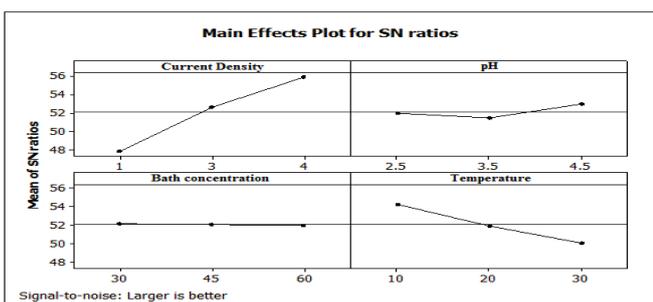


Fig. 6: S/N ratio plot for means of S/N ratio

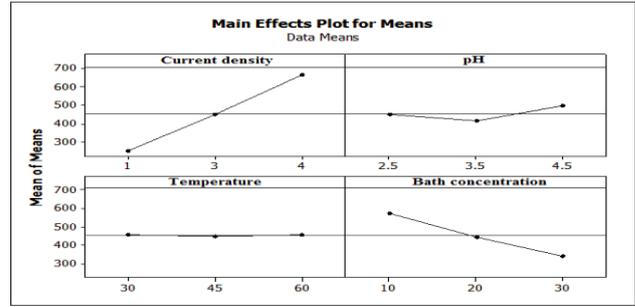


Fig. 7: Mean effect plot for means

VII. ANALYSIS OF VARIANCE (ANOVA)

ANOVA is a collection of statistical models used in order to investigate the differences between the group means and their related procedures such as variation among groups. In this study, a particular variable is divided into components available to different source of variation. This is done by calculating the variation of the S/N ratios (sum of the squared deviations from the total mean S/N ratio) into contributions by each process parameter and error. The percentage contributions of variance are calculated by the subsequent equations. The total sum of the squared deviations (SST) from the total mean S/N ratio can be expressed as;

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

Where "n" is the number of experiment in the orthogonal array, η_i is the S/N ratio of the i^{th} 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 the sum of the squares of deviation.

F-test is a statistical tool (the mean square error to residual) in ANOVA used to find out the most significant parameters that influence the quality characteristic. Higher the F-value will be most influential on the response quality characteristic P-value demonstrate the significance level (significant or non-significant) of the process parameter.

Table V: ANOVA table for micro hardness

Source	DF	Seq SS	F	P	ρ %	Si g
a.	2	76945 4	20.4 6	0.00 0	55.4	1
b.	2	32392	0.86	0.43 9	2.34	3
c.	2	319	0.01	0.99 2	0.02 3	4
d.	2	24484 8	6.51	0.00 7	17.6 8	2
Error	18	338490				
Total	26	138550 4				

Table V gives the results of ANOVA for micro hardness. From table V, it is observed that the most significant

parameter that influence microhardness of the coating are of order of current density, ρ % (55.4); bath concentration, ρ % (17.68%); pH of bath, ρ % (2.34); and temperature of bath, ρ % (0.023).

VIII. CONCLUSIONS

Nickel-SiC + Flyash electrodeposited composites have been produced from watts bath using conventional. The experiments were carried out using Taguchi approach with the objective of less experimental trails and cost effective experimentation. Greater micro hardness has been attained in Ni- SiC + Flyash deposits than pure nickel. The following conclusions were established from the experimental and studies.

- SiC + Flyash particles after the ball milling process (for homogeneity) were successfully dispersed in nickel matrix by conventional type electro depositions.
- L27 orthogonal Array of Taguchi's approach was used to frame the experimental trails with minimum number of experiments. The experiments were conducted by adjusting the process parameters and levels based on the run orders.
- The surface morphological studies for Ni- SiC + Flyash coating was performed using optical micrographs and SEM analysis in order to confirm the particle deposition in Ni matrix. With these, EDAX investigations were performed to in order to confirm presence of SiC-Flyash in nickel matrix.
- Experimental outcomes such as mass of deposit, coating thickness and micro hardness of coating were investigated systematically.
- The influences of process parameters on the response micro hardness were studied via analytical studies such as mean effect studies and S/N ratio analysis. In order to confirm the end results of above analysis, significance analysis studies were conducted using statistical tool called ANOVA. The significances of process parameters were ranked by order.
- From the above investigations, current density and concentration of the bath are the most significance factor for the hardness for conventional type deposition.
- It is important to note that the mass of deposit was accomplished from 53.2 to 314.87 mg. The micro hardness of Ni- SiC+Flyash coating was obtained in the span of 215 to 1072 HV.
- The influences and significances of process parameters on micro hardness of coatings were investigated and rank orders were proved with scientifically and statistical methods and improved the trustworthiness of experimental investigations.

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