Experimental Investigations and Effect Studies on Electrodeposited Nickel-Slag Powder Composite Coatings

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Abstract

Objectives: This paper presents an influences of process parameters on micro hardness of electrodeposited Nickelslag powder composite coating based on Taguchi's experimental design, signal to noise ratio and analysis of variances. **Methods**: Nickel – slag powder composite coatings are produced by conventional type electro deposition on mild steel substrate. The primary parameters; current density, pH, bath temperature, particle concentration and agitation speed are considered for experimental investigations. The experimental design is framed by L₂₇ orthogonal array of Taguchi's model based on five parameters with three levels. The experiments are conducted in Watt's type nickel bath. **Findings**: The micro hardness values of coating were determined using Vickers micro hardness tester. The micro hardness of the Ni-Slag coating was obtained in the span of 190 to 358 Hv. The significances of process parameters on micro hardness of coating were investigated with signal to noise ratio and analysis of variances, and ranked by order. It is observed that bath concentration and agitation speed are the most influencing factors on micro hardness. **Application**: Taguchi's experimental approach reduces the experimental trails and contributing a greater advantage with reduced experimental time and cost of experimentation. Statistical investigations extend the reliability of an experimental work instead of conventional and randomized testing procedures.

Keywords: Analysis of Variance, Composite Coating, Electrodeposition, Microhardness, Orthogonal Array, S-N Ratio

1. Introduction

Electrodeposition process has been acknowledged technique for fabrication of micro and nano crystalline composites. This technology is feasible for many applications and economically superior technique for manufacture of metallic and composite coatings¹. Electrodeposited composite coatings consist of a matrix prepared by metallic compounds and codepositing materials (second phase material) can be a metallic or nonmetallic powder, encapsulated particles or fibers. In electrodeposition or electro-codeposition of composite coating, insoluble reinforcing elements are held in suspension in a conventional electrolytic bath. During plating the reinforcement particles are codeposited in the growing metal matrix layer by electroplating principles. The objective of developing composite coating is to enhance one or more of its properties or to obtain an entirely new property over a substrate material. Mostly the electrodeposited composite coatings are prepared to

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increase the surface hardness of substrate material and wear resistance characteristics. Ceramic particles such as Al₂O₃, cermets, MoS₂, SiC, Si₃N₄ and TiO₂²⁻⁷; metal particles such metal particles such as Al, Cr, Cu, Mo, Ti, V and W⁸⁻¹¹; PTFE¹²; diamond¹³; pumice¹⁴ are successfully co-deposited in various metal and alloy matrices with the help of electro co-deposition practices by various researchers for the past three decades. Numerous experimental investigations are mainly focused on parameters such as current applied, pH of the bath, bath temperature, particle concentrations and agitation of the bath for preparation of electrodeposited composite coating. Ni-SiC composite coating is prepared in watts bath by conventional and sediment codepostion methods and Micro and Nano sized SiC particles were employed as reinforcing elements, and revealed that the sediment codepostion method produced substantial improvement in microhardness and wear resistance⁵. The enhancement of Vickers micro hardness of Ni-Al particles composite coating were amplified with plating parameters such as current density and particle concentration in bath¹⁵. Ni - pumice cost effective coating is developed via elecrodepostion technique by considering current density, pH, temperature and stirring speed were the major experimental factors on microhardness and wear resistance behaviors14. Nickel + Titanium, Nickel + Aluminum and Nickel + Titanium + Aluminum coatings are prepared through electrodeposition and revealed that the deposits produced uniform implantation of Al and Ti particles in Ni matrix. Also recognized that the metal powder amount and current density were took prime task in particle deposition into the matrix¹⁶.

Statistical forecasting models for various performances in terms of process parameters are investigated by numerous researchers and also established the best levels of the process parameters. Different statistical methods that are available to investigate the influences of process parameters in Design Of Experiments (DOE) such as one fractional, full factorial and robust design of experiments. In addition, better quality of the experimental procedure is the primary principle of the robust design method and minimizes the cause discrepancy effects without negotiating the roots⁴. Design Of Experiments (DOE) methodology is employed in numerous engineering applications to minimize cost and time. This technique be capable of characterize and investigate all the process conditions and factors involved in the experimental work. In DOE approach, experimental consequences are analyzed by statistical techniques and the significance of process parameters on experimental outcomes can be estimated¹⁷.

The effects studies on Ni - YSZ composite coating was implemented using L9 orthogonal array of taguchi approach and S/N ratio analysis. Current density, particle concentration and time of deposition on area fraction of YSZ particles were considered on micro hardness and thickness of Ni-YSZ composite coating. The experimental outcomes were analyzed with S/N ratio and ANOVA studies, and process parameters were ranked by order based on the influences¹⁸. The prediction models obtained by them were also good agreement with the experimental results¹⁸. Electro less Ni-B composite coating was prepare and the influences of process parameters were investigated on microhardness of coating with the help of taguchi analysis. Four parameters, namely, bath temperature, concentration of nickel source, concentration of reducing agent and annealing temperature were considered, and framed into an L27 orthogonal array. The significances of process parameters on hardness of the coating were determined with the help of analysis of variance, which revealed that annealing temperature and concentration of reducing agent have significant influence on hardness characteristics of electroless Ni-B coating¹⁹. With the above instances, statistical prediction models have applied in various field investigations to evaluate the best levels of the process parameters by many researchers²⁰⁻²².

However, previous experimental studies in composite coatings were accomplished by randomized manner and only few process parameters were considered in statistical studies in identify the influences on microhardness, volume fractions and coating thickness. Selection of process parameters also has not been done in proper categorization. In this article, five principal process parameters of electrodepostion process such as current density, pH of bath, bath temperature and slag particles concentration in the bath and agitation speed are considered on micro hardness of Ni-slag powder coatings. It is a new approach, recycling of slags for preparation of composite coating, taken from molten metal of aluminum. It is also a cost effective technique by utilization of foundry wastes for composite coating fabrications. The slag flakes were crushed to micron sized powders applied as a reinforcing element in Ni matrix. The compositions were investigated with XRD analyzer, revealed that slag material composed with imperative ceramic phases. In order to indentify the compositions and phases in the slag, X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) analysis were conducted. The

elemental compositions and phases of slag power are summarized in Table 1 and Figure 1. It was decided that utilization of those ceramic phases for composite structure preparation. The Taguchi method of L27 orthogonal array has been employed to study the influences of process parameters on microhardness of Ni – slag power coating. With these, Analysis of Variance (ANOVA) techniques has been applied to determine the significance of these parameters.





2. Taguchi Method

Many of experimental works have to be conducted to learn the characteristics of the response influenced by various process conditions. Taguchi technique is an experimental design technique applied for engineering analyses to optimize the levels of process parameters for the required performance characteristic. This methodology reduces the number of experiments by establishing a special design of orthogonal arrays. The orthogonal array approach facilitates to study the entire parameter space with minimum number of experiments. Thus, it reduces time and cost of the experiment. Taguchi approach employs loss function to determine the performance characteristic conflicting from the desired value. The loss function value is switchover into signal-to-noise (S/N) ratio. The phrase "signal" stands for the desirable (mean) values, and the term "noise" represents the undesirable (standard deviation (SD)) values for the output characteristic.

The objective of the present work is to maximize the micro hardness of the Ni- slag powder composite coatings; hence the higher is the better module is tailored. The higher is better characteristic S/N ratio can be formulated as;

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

Where n is equal to replication of the experimental work and y represents the output of experiment. In addition to S/N ratio, a statistical technique, ANOVA can be utilized to determine the influence of the process parameters on the performance characteristic.

Hence, the above mentioned aspect are the motivations for this study to investigate the influence of process parameters on micro hardness of Ni-slag particles composite coating using Taguchi's orthogonal array studies. L27 orthogonal was employed for conducting the experiment. The process parameter and their level are shown in Table 1.

	Fable 1.	Elemental	compositions	of slag	powde
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XRF – Results						
Elements						
Specification	Observed value %					
Fe	2.24					
Co	0.31					
Zn	0.06					
Мо	0.01					
LE	95.07					
Cd	2.31					

 Table 2.
 Process parameters and levels

	TT •4	Levels			
Parameters	Units	Ι	II	III	
a. Current density	A/dm ²	1	2	4	
b. pH of bath	pН	2.5	3.5	4.5	
c. Temperature of bath	°C	30	45	60	
d. Bath concentration	g/l	10	20	30	
e. Agitation speed	rpm	200	250	300	

3. Experimental

3.1 Preparation and Characterization of Slag Powder

Slag flakes were obtained from foundry shop while melting of aluminum, was crushed to micron sized powder using mechanical type pulvarizer. In order to reduce the size of particles further, ball milling was employed for 8 hrs with 100 rpm with the assistance of ceramic balls. The obtained powder was washed in distilled several for minutes for removal impurities and dust contents. In addition the powder was heated up to 100 °C for removal of moisture content. The cleaned and purified slag particles with 8µm average sized were taken for experimental study.

3.2 Materials Used

A watts type nickel bath⁵ was employed for deposition work. The nickel electrolyte medium was prepared from laboratory graded and purified by conventional method. 1000 ml of solution was taken for plating work in 2000 ml BOROSIL glass beaker and the pH of the solution was adjusted to 5 initially.

A pure nickel plate of size $102 \times 43 \times 5 \text{ mm}^3$ was employed as anode for Ni metal matrix formation. A cold rolled mild steel plate of size $71 \times 25.4 \times 1.2 \text{ mm}^3$ was used as cathode substrate. The mild steel cathode plate was degreased with acetone and polished using dry cloth buffing wheel, for amputation of rust layer. The mass of each mild steel plate was weighed before plating using electronic balance. Effective area of deposition was taken as $25.4 \times 25.4 \text{ mm}^2$ for plating work on polished surface and the remaining portions of cathode plate were masked. Fine particles of slag powder with required quantity was appended to the solution. The plating solution along with slag powder was stirred for 3 hours before plating for getting of homogeneous blend along with surfactant to ensure the co deposition. Each mild steel cathode plate was etched in cathodic and anodic cleaning bath for removal oxide contamination in plating area and to confirm better adhesion of coating and finally rinsed with distilled water, and kept immersed in plating bath.

The reinforcing particles were kept in suspension via mechanical agitation using a motorized stirrer. Agitation speed of the Stirrer was monitored by digital tachometer and attuned with speed controller. A regulated D. C. power supply machine (made by Spark Tek, India, Capacity: 0 - 30V and 0-2 A) was employed for electrodeposition. The bath pH was observed by digital type 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 using 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 maintained constantly. Both plates were vertically positioned for all experiments. This plating technique is called conventional type electrodeposition (CED) technique²³. The CED type electrodepostion setup is shown in Figure 2. The run orders of parameter levels given in Table 3. The experiments were performed based on the run orders of L27 orthogonal pattern. The plating Vol 8 (22) | September 2015 | www.indjst.org

parameters were precisely controlled during deposition and the experiments were conducted at normal pressure levels. The time extent for each of plating was taken as 60 min for all cases.



Figure 2. Electrodeposition setup.

3.3 Formation of Ni – Slag Particle Composite Coating

The principle of electro co-deposition is identical to the fundamentals of electroplating. Addition of reinforcing elements such as ceramic, inert particles or metal powder into the electrolytic bath is the most important task in electro co-deposition. In this study, slag particles were assorted in Watts nickel bath and kept in suspension by mechanical agitation. During plating the charged nickel ions which released from Ni-anode captured the slag particles kept suspension in the bath and deposit the same into the cathode substrate. The continued incident of this phenomenon directs the formation of metal matrix composites.

3.4 Assessment of Surface Morphology, Volume Fraction and Microhardness of Ni – Slag Powder Composite Coating

The coated samples were prepared for surface morphological examinations via metallographic procedures. Initially, the deposition nature and distributions of slag particles in nickel matrix were absorbed with high transmission Trinocular metallurgical microscope (KYOWA, model ME- LUX2, 50x-1000x, Japan) on different magnifications.

The Area and volume fractions of slag particles in the matrix were examined via CCD camera (WATEC, model WAT-221S, Japan) and image analyzer system. Figure 4 (a) and (b) shows the distribution of slag particles in nickel matrix captured in the optical CCD camera. Surface morphologies of Ni – slag composite coatings were investigated with the assistance of Scanning Electron Microscope (JEOL–Field emission SEM, model TSM-6701F, Japan) at various magnifications and shown in Figure 3 (a) and (b). It was observed that the particles were uniformly deposited in matrix and authorizes the particle deposition. Microhardness of the coated sample was inspected in Vickers micro hardness tester (model & maker: SHIMADZU - TYPE HMV - 1/ - 2, SHIMADZU Corporation, Japan) with the payload of 150 gm for 10 sec of indentation period. The indented location was focused at 400X magnification and the slider position was attuned to the diagonal lengths of indentation. Micro hardness was calculated by a system based on and value was taken from digital read out. Micro hardness of each sample was inspected with three trials and the average value taken for reporting. The micro hardness of Ni – slag composite plating attains maximum 358 HV in comparison with 198 HV of pure Ni coating²⁵.



Figure 3. SEM micrographs of Ni – Slag coating – Magnifications: **(a)** 500X and **(b)** 1000X.

 Table 3.
 Process parameters and levels

Expt. C		Contr	ol Parar	neters		Mass of Deposit	Vol. Fraction (%)	Micro Hardnes	S/N ratio (dB)
No.	a	b	с	d	e	(mg)		(HV)	
1	1	1	1	1	1	77.7	24.75	259	48.25
2	1	1	1	1	2	54.6	16.36	271	48.65
3	1	1	1	1	3	27.8	10.21	258	48.23
4	1	2	2	2	1	84.5	23.33	227	47.12
5	1	2	2	2	2	98.1	25.11	198	45.89
6	1	2	2	2	3	85.5	18.30	274	48.75
7	1	3	3	3	1	55.8	23.14	285	49.09
8	1	3	3	3	2	97.6	11.26	246	47.81
`9	1	3	3	3	3	73.3	16.16	254	48.11
10	2	1	2	3	1	127.7	25.28	263	48.39
11	2	1	2	3	2	311.7	16.16	237	47.48
12	2	1	2	3	3	219.9	24.75	358	51.07
13	2	2	3	1	1	99.2	23.86	269	48.59
14	2	2	3	1	2	149.8	26.49	278	48.87
15	2	2	3	1	3	395.9	22.96	231	47.27
16	2	3	1	2	1	58.1	10.84	233	47.33
17	2	3	1	2	2	33.7	9.78	190	45.59
18	2	3	1	2	3	39.8	10.42	201	46.06
19	3	1	3	2	1	374.5	24.75	198	45.92
20	3	1	3	2	2	355.5	21.14	192	45.65
21	3	1	3	2	3	281.7	25.63	314	49.95
22	3	2	1	3	1	64.5	27.35	306	49.70
23	3	2	1	3	2	93.2	15.36	258	48.24
24	3	2	1	3	3	101.6	22.06	242	47.66
25	3	3	2	1	1	322.3	26.49	275	48.80
26	3	3	2	1	2	351.9	15.16	266	48.49
27	3	3	2	1	3	303.1	22.24	245	47.77

4. Results and Discussion

4.1 Analysis of S/N Ratio

It is essential to investigate the S/N ratio factor¹⁸ from the experimental statistics to compute the average S/N ratio response for each experimental factor. From the mean S/N response factor, the most favorable plating conditions for each design parameters can be identified and the process parameters can be ranked according to their impact on the response parameter. In this experimental design, micro hardness of deposit is the response variable which needs to be maximized and hence larger the better characteristics was preferred for this experimental investigations. 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 experiment outcomes and S/N ratio values for microhardness are given in Table 3. Table 4 indicates the mean of the response variable (micro hardness) for each level of each control factor. The mean response table 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 maximum difference will give the most significant parameters, and rank for the significant parameters is depicted. Mean effect plots for process parameters are given in Figure 4. The same procedure is applied for S/N ratio response for each level of process parameter, and S/N ratio response for micro hardness is given in Table 5.

 Table 4.
 Mean response table for microhardness

LEVEL	a.	b.	с.	d	e.
1	252.20	260.9	246.3	261.20	257.00
2	251.00	253.5	260.10	225.10	237.10
3	255.00	243.8	251.8	271.90	264.10
Max-Min Δ	4.00	17.1	13.7	46.90	27.00
RANK	5	3	4	1	2



Figure 4. Mean effect plots for factors.

The consequences of mean S/N ratio response values are shown in Table 5. 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 the bath concentration has the highest delta value, ranked by 1st position and identified as the most influencing factor on micro hardness. The other factors such as agitation speed, pH factor, temperature and current density were ranked by order.

Table 5. S/N ratio table for microhardness

LEVEL	a.	b.	с.	d	e.
1	47.99	48.18	47.75	48.33	48.13
2	47.85	48.01	48.20	46.92	47.41
3	48.02	47.67	47.92	48.62	48.32
Max-Min Δ	0.17	0.51	0.45	1.70	0.91
RANK	5	3	4	1	2

4.2 Analysis of Variance (ANOVA)

ANOVA is a statistical based, objective decision making tool is employed to find out the influence of process parameters on quality characteristics. It gives a hand in testing the significance of all process parameters by evaluating the mean square against an estimate of the experimental error at specific confidence levels. This is carried out by calculating the variability 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 computed by the following equations. The total sum of the squared deviations (SS_T) from the total mean S/N ratio can be expressed as;

$$SS_{T} = \sum_{i=1}^{n} (n_{i} - n_{n})$$
(2)

Where 'n' is the number of experiment in the orthogonal array, ' η_i ' is the S/N ratio of the ith experiment and ' η_n ' is the total mean S/N ratio. The percentage contribution of variance (ρ) can be calculated as follows;

$$\rho = (SS_{\rm p} / SS_{\rm T}) \tag{3}$$

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 applied to investigate the most significant process parameters that influence the quality characteristic²⁴. Higher the F-value will be most influential on the response quality characteristic. P-value demonstrates the significance level (significant or non significant) of the process parameter.

Table 6 gives the results of ANOVA for micro hardness. It is observed that the most significant parameter that influence microhardness of the coating are in the order of bath concentration, (27.44%); agitation speed, (8.79%); pH of bath, (3.39%); temperature of bath, (2.29%); and current density, (0.18%).

Factor	DF	SS	F	Р	ρ%	Sig
a.	2	75	0.03	0.974	0.189	5
b.	2	1343	0.47	0.634	3.394	3
с.	2	882	0.31	0.739	2.229	4
e.	2	10856	3.79	0.045	27.442	1
d.	2	3480	1.21	0.323	8.796	2
Error	16	22923				
Total	26	39559				

5. Conclusion

Recycling and codeposition of newer material, slag powder were successfully deposited in to nickel matrix. It is a cost effective technique and novel approach to develop a metal matrix composites by using industrial squanders. The following conclusions were established from the experimental and analytical studies.

- L27 orthogonal array of Taguchi's approach was implemented to frame the experimental trials with minimum number of experiments. The experiments were conducted in watts bath and the parameters were controlled precisely.
- The surface morphological studies for Ni slag composite coating was performed using optical micrographs and SEM analysis in order to confirm the particle deposition in Ni matrix.
- Experimental outcomes such as mass of deposit, volume fractions of particles and microhardness of coating were investigated methodically.
- The influences of process parameters on microhardness were investigated 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 by a statistical tool called ANOVA.

- The results and rank orders computed from three analytical modules revealed that the bath concentration is the most significance factor for the response.
- Such statistical explorations develop the reliability of an experimental work instead of conventional and randomized testing procedures. It is also reducing the time and cost of experiments.
- It is important to note that the mass of deposit was accomplished from 27.8mg to 395.9mg, volume fraction of particles found to be 9.77 to 27.34% and the microhardness of the Ni-Slag coating was obtained in the span of 190 to 358 HV.

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