# Influence of process parameters on Electrodeposited Nickel thin film coating using Taguchi approach

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*Abstract*— **In this study, Nickel thin film coatings were prepared from watts bath with the aid of electrodepostion process over a mild steel substrate. The process parameters current density, pH, bath temperature and time of deposition were considered for experimental work. The L27 orthogonal array of taguchi design was chosen for experimental design. Based on the run orders of experimental design the experiments were conducted. Micro hardness of thin film coatings were examined in Vickers micro hardness tester under the pay load of 50 gram force. The surface morphologies of coatings were investigated with scanning electron microscopic analysis and the deposition of nickel were confirmed by EDX analysis. The influences of process parameters on micro hardness of nickel coatings were investigated with S/N ratio analysis and mean effects studies via Taguchi approach and parameters were ranked by order. It is observed that current density and time of deposition were most influencing factor for micro hardness of Nickel coating. In order to confirm the rank positions, Analysis of variances (ANOVA) test were conducted and resulted the similar rank positions of S/N ratio and mean effects studies.**

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

## I. INTRODUCTION

Electrodeposition has been acknowledged as a feasible and cost effective technique for producing of thin film coating. Electrodeposited materials are possessed with optimal mechanical properties and suitable for fabrication of micromechanical components with better properties than that of conventional type fabrication. Numerous metals or alloys can be electroplated with different properties can be attained. Electroplating is well-suited with integrated circuits production as it is low-temperature and high rate deposition technology.

Nickel is an extensively applied material for thin film coating by electrodeposition. The nickel thin film coatings were prepared by different grain structures by above process. Large grained Ni coating is anticipated to distort easier whereas fine-grained structured electrodeposited nickel will resist the deformation. Electrodeposited nickel possessed with good mechanical properties such as high yield strength and hardness that are advantageous with the elevated microstructures. Nickel plating is very important for a microelectromechanical systems (MEMS) device with a better electrical and thermal conductivity and also important for applications like small integrated systems with sensors, signal conditioning circuits, actuators and with special functions with a size of few micrometers [1].

Zhao et al [2] had developed a nickel thin film in sulphamate bath at different current densities ranging from 0.01 $A/cm^2$ to 0.1 $A/cm^2$. They investigated the microstructure and corrosion behaviors of electrodeposited nickel developed at different current densities. It was indentified that during electrodeposition grain size increased with increasing current density, the dense surface microstructure of electrodeposited nickel was developed at $0.05 A/cm^2$. The prepared nickel samples possessed with active–passive–trans passive polarization properties. Better corrosion resistance in nickel plating was obtained at 0.05 $A/cm^2$ of current density.

Arman zarebidaki [3] had prepared electrodeposited nickel coating onto AZ91 Mg alloy. They concluded that depositing a pore-free nickel coating with average grain size of 95 nm can be accomplished by using proper initiation, zincating and Cu electrode-position pretreatment processes. The nickel layer developed in this condition can efficiently improve the corrosion resistance and hardness of AZ91 Mg alloy that makes it more dependable for industrial applications. Zengwei Zhu [4] had prepared bright nickel electro deposited coating under perturbation of hard particles. They found that bright nickel deposit was FCC structure. The surface roughness was about 0.016 µm and the grain size was less than 80 nm.

Chemical contamination of electrodeposited coatings must be carefully assessed before discussing the influence of their microstructure on properties [5]. ZHOU Li-qu et al [6] had equipped the forming limits of nickel coating. They concluded that the forming limit of the nickel coating is not as good as that of the steel substrate, and the forming limited strain of the coating sheet tends to moderate with the rise of thickness of the coating.

A- Ul-Hamid et al [7] had prepared electro deposited Nickel coating on Al 2014 alloy. This experimental result indicates that electrochemical deposition combined with heat treatment can be used to improve the surface properties of Al alloy. Anette A. Rasmussen [8] concluded that the current density and the temperature only have a limited effect on the microstructure and the hardness.

Suman and sahoo [9] had prepared the electroless Ni-B composite coating and investigated the influences of process parameters on microharness of coating with the help of taguchi analysis. They took four parameters; temperature of bath, nickel source concentration, concentration of reducing agent and annealing temperature, and framed into an L27

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orthogonal array to determine the significances of process parameters on hardness of the coating with the help of analysis of variance, which exposed that annealing temperature and concentration of reducing agent have significant influence on hardness characteristics of electroless Ni-B coating. With the above illustrations, statistical prediction models have applied in various field investigations to evaluate the best levels of the process parameters by many researchers [10-12].

However, previous experimental studies in electro deposition were accomplished by randomized manner. 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, four principal process parameters of electrodepostion process such as current density, pH of bath, time of deposition and bath temperature are considered on micro hardness of Ni thin film coatings. The taguchi method of L27 orthogonal array has been employed to study the influences of process parameters on microhardness of Ni thin film coating. With these, Analysis of Variance (ANOVA) techniques has been applied to determine the significance of these parameters.

## II. TAGUCHI APPROACH

The scientific approach to quality improvement was becoming more widespread in industrial practice. The application of statistical methods, in particular the design of experiments, has had considerable impact. The ideas of a very successful leading quality consultant in Japan, Dr. Taguchi have been adopted by many American companies in both manufacturing and scientific circumstances. The experimental designs developed by taguchi known as orthogonal arrays, are essentially fractional factorials.

Knowing the quantity of parameters and therefore the number of levels, the appropriate orthogonal array is designated. During this work we have a tendency to seized four plating parameters with 3 levels. Supported the on top of parameters and levels, L27 orthogonal array of Taguchi approach was executed for sturdy experimental style. The target of the present work is to maximize the small hardness of the Nickel thin film coatings. Meanwhile the larger is best module is customized .The higher is best representative of S/N ratio can be framed as

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

Where n is cherishing replication of the experimental work and y represents the output of experiment. Additionally to mean effects techniques and ANOVA will be enforced to see the influence of the method parameters on the performance characteristic.

## III. MATERIALS AND METHODS

Electrocodeposition experiments were conducted in a 1000 ml glass beaker. The electrolyte used for plating work was a watts type nickel bath. Mild steel plate of sized 70 × 25.4 × 1 $mm^3$ was used as a cathode substrate and area of deposition taken as 25.4× 25.4$mm^3$ (1 $inch^2$). The left behind portions of plating area were masked. A pure nickel plate was used as anode. A Nickel anode plate of sized 95× 35 × 8 $mm^3$ thick was employed in the circuit. The mild steel cathode plate was degreased by acetone and polished with dry cloth buffing wheel, for exclusion of rust layer. The distance between Ni anode and mild steel cathode was engaged constantly [14]. A regulated power supply unit made by Spark Tek (0-2A, 0 – 30V, DC supply) was used for the electroplating. The bath temperature was accurately controlled using K-type thermocouple. The pH of the solution was monitored by digital pH meter. The pH of bath was attuned with diluted $H_2SO_4$ and NaOH solutions depending upon the plating conditions. The plating conditions taken for these experimental tests are given in table I. The experiments were performed based on the run orders of L27 orthogonal array pattern and twenty seven samples were prepared from the bath.

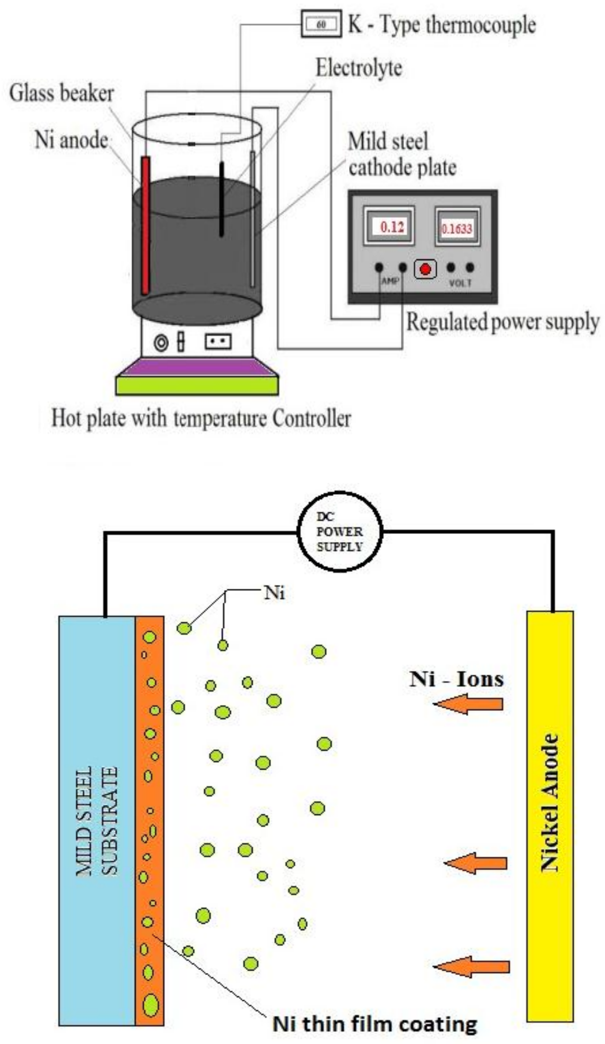


Fig 1: Formation of electro deposited coating

Table I: Parameters and levels

| Parameters | Units | Levels | | |
|---|---|---|---|---|
| | | I | II | III |
| A. Current density | A/dm$^2$ | 1 | 2 | 4 |
| B. pH | | 2.5 | 3.5 | 4.5 |
| C. Time of deposition | min | 30 | 45 | 60 |
| D. Temperature of the bath | $^o$C | 30 | 45 | 60 |

## IV. RESULTS AND DISCUSSION

### A. Assessment of surface morphology

The coated samples were organized for surface morphological examinations via metallographic procedures. The pattern of deposition of metallic element particles within the deposit was examined by scanning microscope (JEOL–Field emission SEM, model TSM-6701F, Japan) at numerous magnifications. From on top of analysis, it absolutely was originate that the particles were uniformly distributed in nickel matrix with uniform grains.

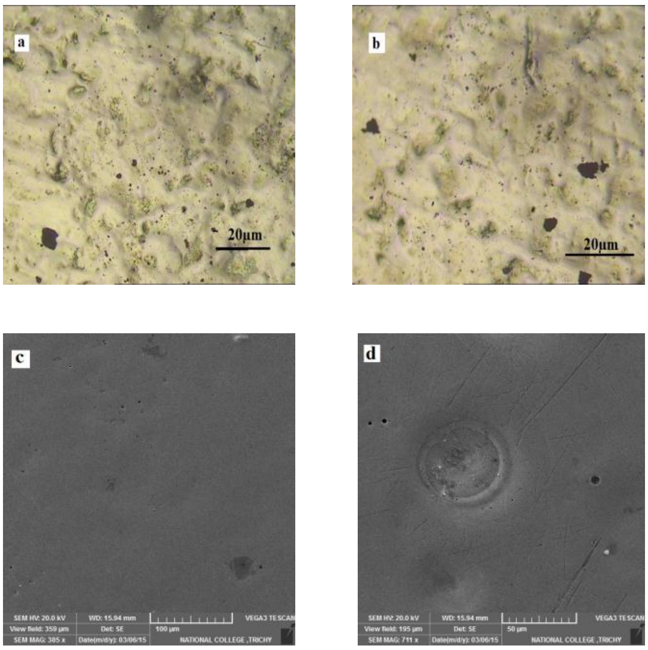


Fig 2: Micro structure of Nickel thin film coating through optical micrographs a.100X; b.250X, SEM micrographs: C. at 385X ; D. 711X

The EDAX observations were accomplished to investigate the compositional phases in the coated samples. Fig 3 displays the EDAX results of composite coating and approves the existence of nickel elements within the matrix. The EDAX observations confirm the presence of nickel elements in the deposition over mild steel substrates.

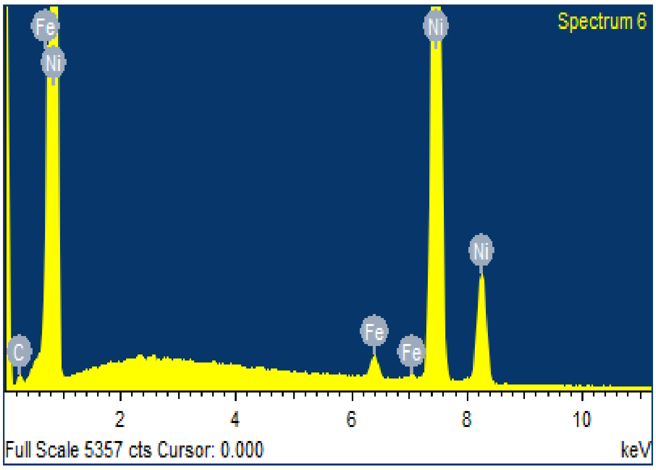


Fig 3: EDX Results of Ni thin film coating

### B. *Assessment of micro hardness of Ni thin film coating*

Micro hardness of the coated samples were discovered in Vickers micro hardness tester (model & maker: SHIMADZU -TYPE HMV- 1/-2, SHIMADZU Corporation, Japan) with the payload of 50 gram force for 10 sec. of indentation amount. The indented location was targeted with 400X Magnification and also the slider positions were adjusted to the diagonal lengths of indentation. Finally, the micro hardness was calculated by a system supported and value was taken from digital scan out. Micro hardness of every sample was examined with four trials and also the average value was taken for final documentation.

### C. Analysis of S/N ratio

Taguchi related with two main parameters called noise and control factors. The control factors are the desirable one that can be controlled and noise factors are undesirable one that amounts for the variation in response characteristics [15]. The process optimization based on the taguchi approach is on ground that the noise factors effect can be reduced if we select proper control factor levels. Larger S\N ratio denotes a higher performance, independent of the operation characteristics and therefore the process can be optimized without removing the cause for variation and making it robust against the noise factors [16].The formulae for signal/noise are designed such the experimentalist can always select the larger factor level settings to optimize the standard characteristics of an experiment. Therefore, the strategy of calculating the signal-to-noise ratio depends on whether the quality representative has smaller-the-best, larger-the-better or nominal-the-better formulation is chosen.

In this study, the quality characteristic has larger-the-best formulation and therefore the equation for calculating S/N ratio is as follows:

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

Where ($y_{ij}$) is the value of the micro hardness for the test in that trial and (n) is the number of tests in a trial, high signal-to-noise ratios are always preferred.

Table II: Experimental responses for Nickel Thin film Coating

| Expt. No. | Control Parameters | | | | Mass of Deposit mg | Micro Hardness HV | S/N Ratio dB |
|---|---|---|---|---|---|---|---|
| | a | b | c | d | | | |
| 1 | 1 | 2.5 | 30 | 30 | 33.2 | 216 | 46.68 |
| 2 | 1 | 2.5 | 60 | 45 | 38.5 | 315 | 49.96 |
| 3 | 1 | 2.5 | 90 | 60 | 62.6 | 399.33 | 52.02 |
| 4 | 1 | 3.5 | 30 | 45 | 10.3 | 231.33 | 47.28 |
| 5 | 1 | 3.5 | 60 | 60 | 59.7 | 340.66 | 50.64 |
| 6 | 1 | 3.5 | 90 | 30 | 72.3 | 474 | 53.51 |
| 7 | 1 | 4.5 | 30 | 60 | 39.5 | 332 | 50.42 |
| 8 | 1 | 4.5 | 60 | 30 | 46.8 | 247 | 47.85 |
| `9 | 1 | 4.5 | 90 | 45 | 53 | 278.66 | 48.90 |
| 10 | 2 | 2.5 | 30 | 30 | 62 | 293.33 | 49.34 |
| 11 | 2 | 2.5 | 60 | 45 | 81.2 | 378.66 | 51.56 |
| 12 | 2 | 2.5 | 90 | 60 | 118.7 | 641.33 | 56.14 |
| 13 | 2 | 3.5 | 30 | 45 | 68.4 | 354.66 | 50.99 |
| 14 | 2 | 3.5 | 60 | 60 | 102.3 | 454.66 | 53.15 |
| 15 | 2 | 3.5 | 90 | 30 | 493.6 | 492.33 | 53.84 |
| 16 | 2 | 4.5 | 30 | 60 | 60 | 277.5 | 48.86 |
| 17 | 2 | 4.5 | 60 | 30 | 98.3 | 277 | 48.84 |
| 18 | 2 | 4.5 | 90 | 45 | 130.8 | 582.66 | 55.30 |
| 19 | 4 | 2.5 | 30 | 30 | 217.3 | 478.66 | 53.60 |
| 20 | 4 | 2.5 | 60 | 45 | 180.4 | 770 | 57.72 |
| 21 | 4 | 2.5 | 90 | 60 | 210.3 | 770.33 | 57.73 |
| 22 | 4 | 3.5 | 30 | 45 | 112.9 | 740 | 57.38 |
| 23 | 4 | 3.5 | 60 | 60 | 157.7 | 683.33 | 56.69 |
| 24 | 4 | 3.5 | 90 | 30 | 246 | 680 | 56.65 |
| 25 | 4 | 4.5 | 30 | 60 | 107.4 | 532.66 | 54.52 |
| 26 | 4 | 4.5 | 60 | 30 | 97.8 | 231.33 | 47.28 |
| 27 | 4 | 4.5 | 90 | 45 | 231.7 | 715.33 | 57.09 |

Table III: Mean S/N ratio values of parameters on micro hardness

| Level | A | B | C | D |
|---|---|---|---|---|
| 1 | 49.70 | 52.76 | 51.01 | 50.85 |
| 2 | 52.01 | 53.35 | 51.53 | 52.91 |
| 3 | 55.41 | 51.01 | 54.58 | 53.36 |
| DELTA | 5.71 | 2.34 | 3.57 | 2.51 |
| RANK | 1 | 4 | 2 | 3 |

Table IV: Mean effects of parameters on micro hardness

| Level | A | B | C | D |
|---|---|---|---|---|
| 1 | 314.9 | 473.6 | 384 | 376.6 |
| 2 | 416.9 | 494.6 | 410.8 | 485.1 |
| 3 | 622.4 | 386 | 559.3 | 492.4 |
| DELTA | 307.5 | 108.5 | 175.3 | 115.8 |
| % of effect of parameters | 43.4 % | 15.3 % | 24.79 % | 16.3% |
| RANK | 1 | 4 | 2 | 3 |

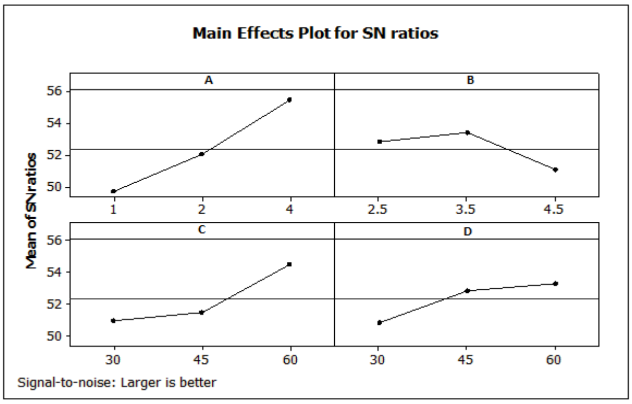


Fig 4: Mean effect plot for S/N ratios

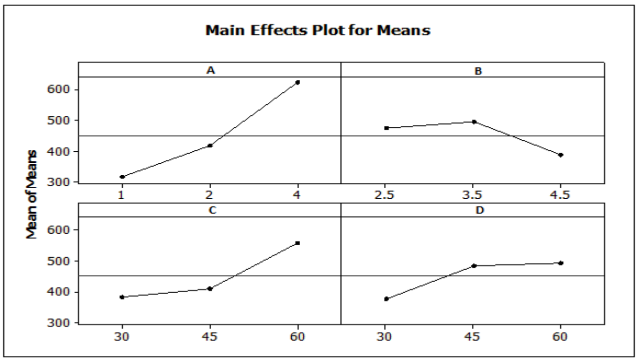


Fig 5: Mean effect plot for means

The significances of S/N ratio and mean effects response were displayed in Tables .This Table also include the delta (Δ) which is the difference among the highest S/N ratio and the lowest S/N ratio values. Ranks for factors are assigned 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 Nickel thin plate electro deposition. The means response table IV designate the percentage of effects of parameters on response (micro hardness), and the rank orders were identical to the rank orders of S/N ratio analysis. In order to confirm the results of S/N ratio and mean effects studies, ANOVA approach was employed to identify the significances of process parameters on response variable.

### *D. Analysis of variance (ANOVA)*

The intention of analysis of variance (ANOVA) is to test for significant differences between means. 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 V: ANOVA table for micro hardness - conventional type deposition

| source | DF | SS | F | p | ρ % | Rank |
|---|---|---|---|---|---|---|
| A | 2 | 441616 | 24.91 | 0.000 | 49.23 | 1 |
| B | 2 | 59681 | 3.37 | 0.057 | 6.65 | 4 |
| C | 2 | 160505 | 9.05 | 0.002 | 17.89 | 2 |
| D | 2 | 75712 | 4.27 | 0.030 | 8.44 | 3 |
| | Error | 18 | 159574 | | | |
| | Total | 26 | 897088 | | | |

Table V shows the contribution levels (**ρ %**) of parameters on micro hardness investigated from ANOVA module for Ni thin film coating electro deposition. It was witnessed that, the contributions of parameters on micro hardness was about current density, ($\rho$=49.23%); time of deposition, ($\rho$=17.89%); temperature of the bath, ($\rho$=8.44%); pH of the bath,(6.65%).The above rank orders are worthy 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 confirmed with three different schemes and authenticated.

## V. CONCLUSIONS

Electrodeposited Nickel thin film coating have been produced from watts bath. 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. The following conclusions were established from the experimental and analytical studies.

- L27 orthogonal array of taguchi's approach was engaged to frame the experimental trails with least number of experiments. The experiments were accomplished by adjusting the process parameters and levels based on the run orders.
- The surface morphological studies for Ni thin film coating was implemented using optical micrographs and SEM analysis.
- Experimental results such as mass of deposit, coating thickness, and micro hardness of coating were examined methodically.
- The effects of process parameters on the response micro hardness were examined 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 time of deposition are the most significance factor for the hardness for Ni thin film coating;
- It is important to note that the mass of deposit was accomplished from 70.6 to 250.2 mg. The micro hardness of Ni thin film coating was attained in the span of 216 to 770.33 HV.

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