Parametric Optimization of Electroless Ni-P coatings using Taguchi method
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Abstract. The present work deals with surface roughness characteristics of electroless Ni-P coatings. The optimised process parameters have been determined based on L27 Taguchi Orthogonal array. Bath concentration of nickel sulphate, concentration of sodium hypophosphite and temperature of bath has been considered as variable in this study. The optimum combinations of process parameters and surface roughness have been considered as response variable of the study. It has been found that 24 g/L of Nickel Sulfate, 25 g/L of Sodium Hypophosphite and 80°C temperature gives the minimum surface roughness of the Ni-P coated sample which was 0.23µm. Characterisation of the substrate and the as deposited samples were done by scanning electron microscope (SEM) and optical microscopy for surface morphology, energy-dispersive X-ray analysis (EDAX) for element analysis and X-ray diffraction for phase analysis.

Keywords: Electroless coating; Taguchi Method; Orthogonal Array

1 Introduction
Electroless Nickel plating is a process of depositing Nickel-Phosphorus coating over a substrate in the absence of electric current [1]. Coatings are deposited over the substrate by chemical reactions taking place in the aqueous solution. Electroless coatings are categorized into pure metal (copper, nickel, etc), polly alloy, nano and composite coatings [2]. The dissimilarity between electroless deposition process and the conventional electroplating processes is that the latter depends on an exterior source of dc in order to reduce nickel ions in the electrolyte to nickel metal on the substrate, while the former does not [3]. The plating bath conditions such as the type and concentrations of the reducing agent, stabilizer, pH and the temperature are governed by the deposition rate, properties of coated components and the structural behaviour of the deposits mainly. The characteristics and microstructures of EN coatings generally depend on the quantity of phosphorous alloyed in the deposit [4]. Due to high porosity the corrosion resistance and surface roughness of the coating is reduced. In general, phosphorus content (1–5%) EN deposits are microcrystalline, medium content phosphorus (6–9%) coatings has mixed crystalline and amorphous microstructures [5].

Surface roughness is an important property where the quality of the resulting surface plays an important role in determining the cost of the product. Like in a textile industry machine, the glazing surfaces of some automobiles [26-27]. Surface roughness has great impact on the mechanical properties like fatigue behaviour, corrosion resistance and creep life [6-9]. As a result, there is a need for development in modelling surface roughness and optimization of the controlling parameters to obtain a surface finish of desired level [10-12]. Thus it is important to optimize the Centre line average roughness (Ra). Several optimization techniques have been used in the past to optimize the input characteristics in order to obtain an optimum response [13-17]. We have applied the Taguchi method for optimization. It uses the concept of orthogonal arrays and Signal to Noise ratio for optimization [18-22]. ANOVA analysis was done to find significant parameters and the significant interactions. It works on the principle of null hypothesis i.e. p value [23-25]. SEM and Optical Microscopy were done to find the surface morphology XRD and EDX was done to find the different phases and weight percentage of each element in the coating.

2 EXPERIMENTAL DETAILS
2.1. Substrate preparation and coating deposition
Copper substrate of size 30x25x1 mm3 is cut from a copper foil (99.0% purity, lobachemie) for the deposition of electroless nickel coatings. The rectangular substrate is cleaned for removing oxide layer
and foreign particles. Firstly the samples are rinsed with 25% HCl solution in 3:1 ratio for 10 min for removing foreign particles and oxide layer. Then, after washing with deionized water, the substrates are dipped into palladium chloride solution (55°C) to activate the surface by overcoming the initial energy barrier and start the deposition process. Then dipping into distilled water for a few seconds, the substrate is ready to be put into electroless bath. This also introduces a significant amount of deposit thickness, higher deposition rates and ensures good adhesion of the coatings to the substrate. Nickel sulphate is used as the source of nickel with sodium hypophosphate as the reducing agent and sodium citrate is added as complexing agent. The coating is deposited over substrate by various steps of chemical reaction. Regarding the Electrochemical reaction, catalytic oxidation of the hypophosphate yields electrons at the catalytic surface, which in turn reduces nickel and hydrogen ions. The chemical concentration of the bath is pre-determined by trial and error method. The pH value of the solution is maintained around 4.5 by continuous monitoring with a pH meter and the temperature of the bath is also maintained by constant supervision with a thermometer. A bath of volume 250 ml is prepared and deposition is carried out for a period of 1 hour. After that, the substrates coated with Ni-P are taken out of the bath and rinsed in distilled water. Then all samples are mounted with the help of epoxy resin. Finally it is ready for testing of surface roughness. Thus it is essential that all samples are of uniform roughness.

2.2 SURFACE ROUGHNESS MEASUREMENT
Surface roughness measurement is done using a portable stylus and skid type profilometer, Talysurf (Taylor Hobson, Surtronic 3+). The profilometer is set to cut off length of 0.8 mm, gaussian filter, and traversed speed 1mm/s with 4mm evaluation length. The measured profile is digitized and processed through the advanced surface finish analysis software Talyprofile for evaluation of the roughness parameters. Talysurf measures the Centre line average roughness. Surface roughness was determined by taking the average of surface roughness recorded over four different places of the coating. Centre line average roughness (Ra) is defined as arithmetic mean deviation of the surface height from the mean line through the profile while the mean line is defined in such a way that it has identical area on either side of it.

2.3 Design of Experiment
Design of experiments (DOE) method is used to reduce the experimental time, number of experiments and computational cost and other resources with the help of conclusive information. Taguchi Optimization method, where orthogonal array (OA) is employed to reduce the number of experiments, is used to determine the optimal process parameters. An OA requires minimum number of experimental trials and provides the shortest possible matrix combination in which parameters are varied to determine the mean data and S/N ratio data through which optimized levels of each factor is determined. In the present investigation, an L27 orthogonal array is used, which has 27 rows and 13 columns where each column represent the factors and their interactions while each row represent the levels of factors and the interacting levels of different factors.

2.4. Taguchi method
Taguchi Modelling is a very effective way to optimize the input parameters in accordance with the output of a signal/experiment with the use of Signal to Noise ratio. The S/N ratio is a ratio of the significant data to the error in an experiment (noise). Hence, a higher value of S/N ratio suggests a better optimization. Hence, the most optimum experiment will have a maximum S/N ratio. S/N ratio works in three different ways according to the response. If the response needs to be optimized in a maxima region then we shall use the ‘Larger the better’ concept of the S/N ratio and if the response needs to be optimized in such a way so that the optimum response is minimum, then we use the S/N ratio which will give ‘smaller is better’. In order to get a nominal value of the response, we shall use the ‘Nominal is best’ formula to get the optimized data. In case of surface roughness we use smaller is the best. Higher S/N ratio designates lesser unrequired noise and more required signal. Therefore a higher S/N ratio suggests a higher optimized results.
3. RESULT AND DISCUSSION

3.1. Surface Roughness

The surface roughness is measured using Talysurf instrument mentioned in the section 2.2. The result obtained from the experiment is given in Table 1.

Table 1: Surface roughness for each coating

| Experiment No | Surface roughness |
|---------------|-------------------|
| 1             | 0.74              |
| 2             | 0.34              |
| 3             | 0.23              |
| 4             | 0.45              |
| 5             | 0.87              |
| 6             | 0.46              |
| 7             | 0.98              |
| 8             | 0.76              |
| 9             | 0.79              |
| 10            | 0.84              |
| 11            | 0.71              |
| 12            | 0.32              |
| 13            | 0.5               |
| 14            | 0.67              |
| 15            | 0.44              |
| 16            | 0.81              |
| 17            | 0.73              |
| 18            | 0.94              |
| 19            | 0.84              |
| 20            | 0.54              |
| 21            | 0.39              |
| 22            | 0.75              |
| 23            | 0.69              |
| 24            | 0.96              |
| 25            | 1.03              |
| 26            | 0.83              |
| 27            | 0.61              |
The surface roughness measured of substrate is calculated at 1.19 µm.

3.2 Taguchi analysis

It works on the basis of the Signal to Noise ratio discussed in section 2.4. The S/N ratio follows a decreasing trend in case of the 3 different levels in the factors Temperature and Nickel Sulphate. From figure 1, it is clearly seen that level 1 of temperature, level 1 of Nickel Sulphate and level 3 of Sodium Hypophosphite are the optimized parameters in order to optimize the surface roughness of the coating, as they have the maximum S/N ratio for that level. The optimized surface roughness is obtained at 0.23 µm.

![Main Effect Plot for SN Ratios](image)

**Figure 1: Main Effect Plot for SN Ratios**

3.3 Characterization

The experimental sample before and after coating are characterised using SEM and Optical Microscopy, XRD and EDX.

![Scanning Electron Microscope Image of Copper Substrate without etching](image)

**Figure 2: Scanning Electron Microscope Image of Copper Substrate without etching**
Figure 2 shows the micrograph of substrate without etching. The micrographs revealed mechanically worked rolled elongated structure. The deposited particles are spherical in nature dispersed over the surface. Figure 3 provide the nature of deposits and its morphology and alignments. The over lapping of particles and its interconnection is clearly visible. Type of deposit is quite dense and firmly attached. The corresponding EDX analysis is indicates the presence of elemental Ni and P in the optimized coated sample. The weight percentage of the elements are Nickel 87.88 and Phosphorus 12.12.

**XRD analysis of Optimize Coated Sample:**

XRD was done for phase determination of the optimised coated sample using X-ray diffraction in Rigaku Ultima- III machine. The range of 2 theta was from 20° to 85° with a scan speed of 2° min−1) in Cu Kα radiation. It can be concluded that compare between copper substrate and as deposite optimised coated sample cu phase converted to micro-crystalline Ni₃P phase which ware shows in below.
Figure 5. X-ray diffraction pattern of optimised as deposited Ni-P coated sample

4. Conclusion
Uniform coating is successfully deposited on the substrates’ surface and Taguchi analysis is successfully applied to find the optimized surface roughness at 0.23 µm with 25g/L Nickel Sulphate, temperature 80°C and 24 g/L of Sodium Hypophosphite. The ANOVA analysis shows that all the 2-way interactions between the three factors, including the Nickel Sulphate factor, are very important in minimising the surface roughness to such low value. EDAX analysis shows the presence of high percentage of Phosphorus which is thus significant in diminishing the surface roughness to a minimal value. The surface roughness of substrate is found to be of 1.19 µm. We have found that decrease in percentage of optimised coated sample to copper substrate is 75.63%.

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