# Study of Nickel Coating on Clay Ceramic Body

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*Clay based ceramic body is successfully coated with nickel using a technique of electroplating. The prerequisite is to convert the surface of the ceramics into electrically conducting state. This can be done through the sensitization step followed by nucleation step and electroless plating process. For this study, the parameters for the sensitization step, nucleation step and the electroplating process are fixed, while changing the parameters for the electroless plating process. Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) studies was employed to examine the quality of the coating. Results show that coating of Nickel was affected by duration and temperature of electroless plating.*

## Introduction

This is a preliminary study to establish an understanding of ceramic electroplating. Over the years, metals and plastics have been successfully electroplated. Based on these experiences it is believed that nickel can be coated on the ceramic surface using the electroplating method. The advantages of coatings made by electrochemical methods of electroplating compared with other coating techniques such as electron beam vapor deposition, thermal spraying and plasma spraying include their versatility, low cost and ability to coat complex shapes. In addition, they can be tailored to meet specific design criteria. Their composition can be varied to enhance properties such as oxidation and thermal resistance and they can be used individually or in multiple layers to enhance the desired performance. Electroplating is a low voltage, high current technique for depositing materials in solution onto a metallic substrate. The electrolytic solution is a conducting fluid in which the flow of current is accomplished through the movement of ions between the anode (positive electrode) and cathode (negative electrode). As the positive metal ions $M^+$ (e.g. $Ni^{2+}$) is reduced at the cathode (substrate) surface, an electroplated coating of the metal M is produced.

Prior to the electroplating process the electroless nickel plating process as discussed by Brenner and Ridell had been utilized to deposit Nickel-Phosphate

(Ni-P) coating onto the surface of clay based ceramic bodies. It is also required that the sensitization step ($SnCl_2$) is followed by nucleation step ($PdCl_2$) before the electroless plating stage begins. SEM was employed to study the dependency of electroless plating parameter on the quality of the nickel electroplating microstructure. Furthermore X-rays diffraction (XRD) technique has been used to analyze the minerals in the samples.

## Experimental

### Sample Preparation

The specimen used was a local clay body with 8% moisture formed at a pressure of 8 tons and sintered at 950°C for one hour. Figure 1 shows the flow chart for the experimental procedures.

The aim of this study is to find the optimum parameters of electroless plating, by varying the range of the water bath temperature from 50 to 95°C and plating time between 10 to 60 minutes. Table 1 shows the electroless nickel plating parameter.

The parameters in the electroplating process were fixed at current of 1 Ampere and plating time of twenty minutes for each sample. Table 2 shows the Watts bath formulation used in this study.

Scanning Electron Microscope Cambridge S200 was used to analyze the microstructures of the nickel coating of each sample after the electroplating process. The X-rays that is used for diffraction are the electromagnetic rays in the range of 0.05 to 0.25 nm. The equipment that is used is PW1729 using the source of $CuK_{\alpha}$ with wavelength 1.5404 Angstrom and the power supply is set at 40kV and 40mA.

## Result and Discussion

Figure 2 shows the microstructure of the surface of the clay sample before the treatment process (pre-plating and plating). It shows that the original sample has many voids, some are small and some are big in size. Porosity level in the specimen was about 30%. Figure 3 shows the result from the SEM showing the microstructures of the nickel coating after the electroplating process. The x-axis shows the plating time and y-axis shows the bath temperature, both for the electroless plating parameters, whereas the parameters for the electroplating process were fixed.

The following discussion is based on the SEM result, referring to Figure 3 the analysis is from the left to the right. At 50°C, the electroless plating solution is not active yet therefore the coating of Ni-P on the sample's surface was not

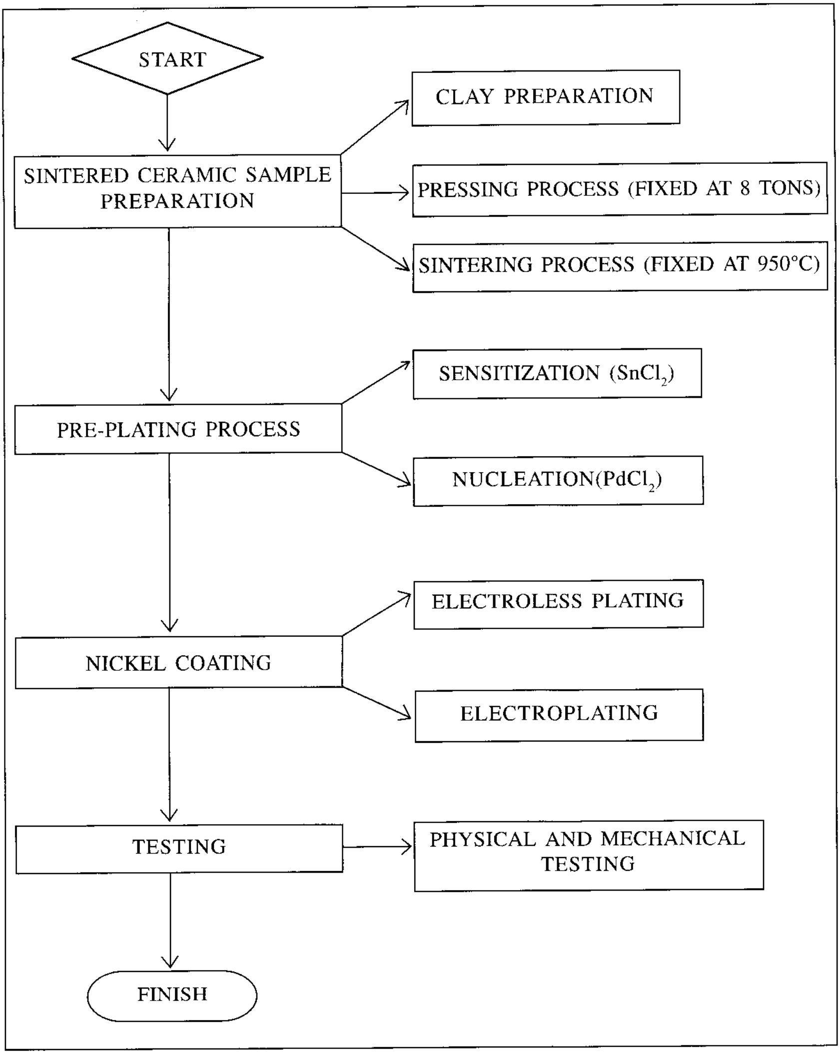


Figure 1: The Flow Chart for Experimental Procedures

Table 1: Electroless Nickel Plating Parameters

| | |
|---|---|
| $NiSO_4.6H_2O:Na_3C_6H_5O_7.2H_2O:NaH_2PO_2.H_2O$ | 11 : 10 : 4 |
| Distilled water | 986.00 ml |
| Bath pH (pH modifier – $H_2SO_4$) | 4.30 (fixed) |
| Bath temperature | (50, 65, 80 and 95)°C |
| Plating time | (10, 15, 30 and 60) minutes |

Table 2: Concentration of ingredients of Watts bath

| | |
|---|---|
| $NiSO_4.5H_2O:NiCl_2.6H_2O:H_3BO_3$ | 80 : 11 : 9 |
| Distilled water | 2 liters |

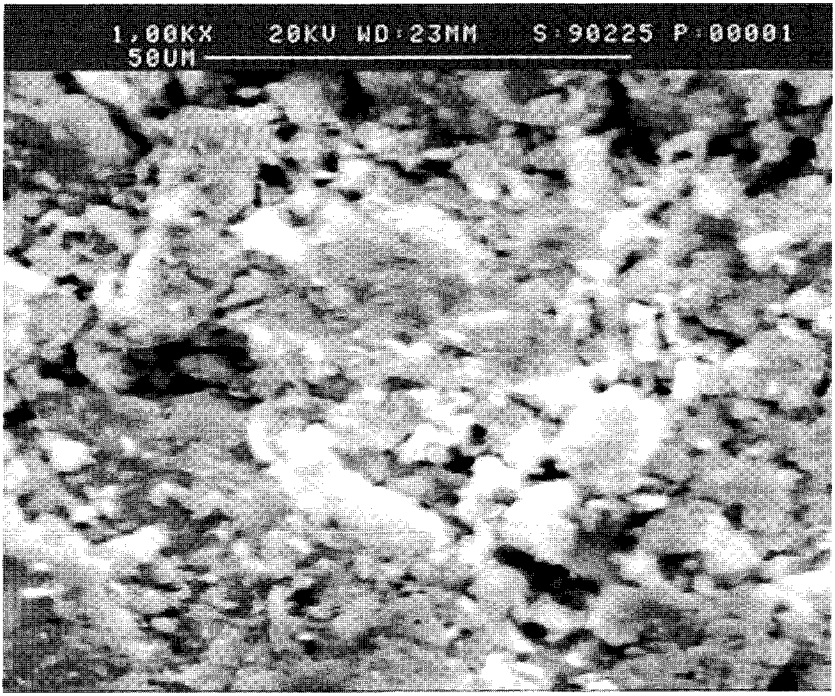


Figure 2: The Microstructures of the Surface of the Sintered Clay Based Ceramic Before the Pre-plating Process.

complete. This results in the poor coating of nickel in the electroplating process. The nickel coating is very thin. The microstructure in Figure 3, for all electroless plating at 50°C are similar to the original sample Figure 2.

At 65°C, the electroless plating solution was active. This resulted in a good coating of Ni-P on the surface of the samples during the electroless plating process. The nickel electroplating process is smoother for all the samples at this temperature. From the figure of the microstructures, it seems that the nickel coating forms islands that gets bigger with the increasing electroless plating time. At the temperature of 80°C and 95°C, the patterns were still the same with exception of 95°C, 60 minutes.

As the electroless plating time increased, the size of the island also increased. Nucleated Ni-P islands acted as catalyst sites for further deposition and therefore

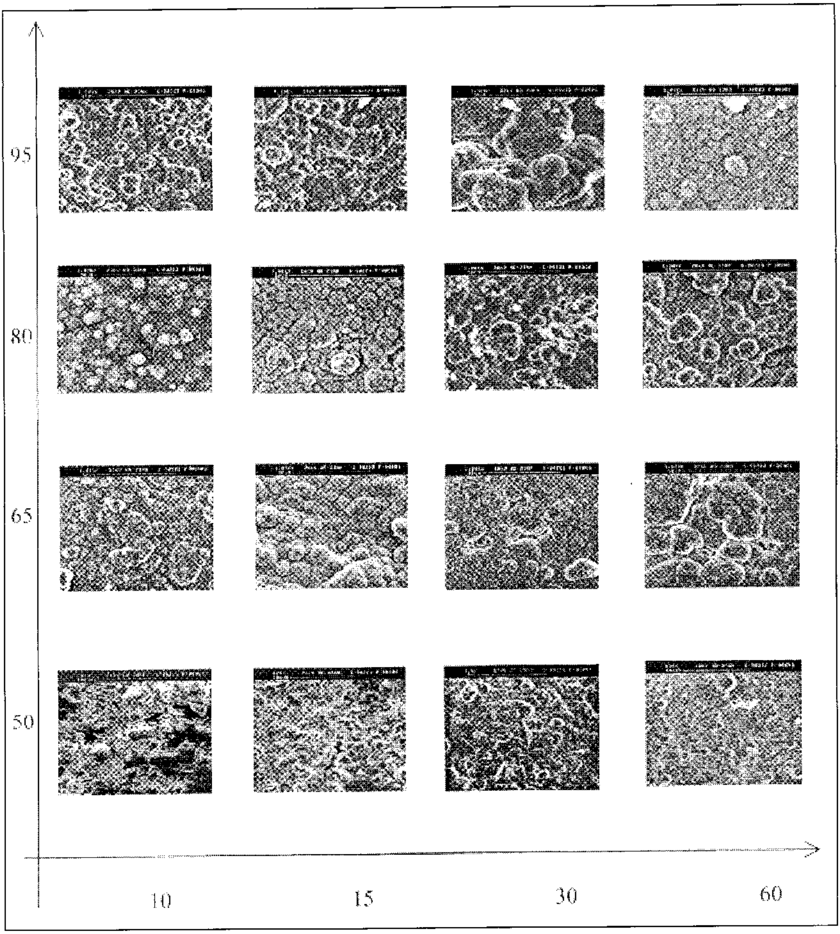


Figure 3: The Microstructures of the Sample's Surface Coated With Nickel After the Electroplating Process. The X-axis Shows The Electroless Plating Time and the Y-axis Shows The Water Bath Temperature for the Electroless Plating Process

resulted in the size increasing with the increasing electroless plating time. But at 95°C, 60 minutes plating time, the nickel islands (to indicate the quality of nickel electroplating) are smaller. It is believed that the higher temperature and longer time resulted in a poor adhesion of the Ni-P that deposited on the sample's surface.

The result also shows that by increasing the temperature, the nickel islands seem to have better adherence. For example, at the 80°C and 95°C temperature, the islands are much clearer, well bonded and more uniform in size compared to the samples prepared at lower temperature of 50°C and 65°C.

Figure 4 until 7 shows result from the X-rays diffraction technique testing that has been done for four types of samples they are clay powder, ceramic plate after the sintering process, ceramic plate after the electroless plating process and ceramic plate after the electroplating process.

Result shows that the clay powder consists of silicate phase ($SiO_2$), kaolinite phase ($Al_2Si_2O_5(OH)_4$)and muscovite phase ($Ka_{12}(Si_3Al)O_{10}(OHF)_2$), Figure 4 is referred. From Figure 5, it shows that ceramic plate after the sintering process consists of silicate phase ($SiO_2$), and mullite phase($Al_6Si_2O_{13}$). Referring to figure 6, the XRD graph analysis shows that the ceramic plate after the electroless plating process consists of silicate ($SiO_2$), and nickel phosphite ($Ni(PO_3)_2$) and from Figure 7, the ceramic plate after the electroplating process consists of nickel (Ni).

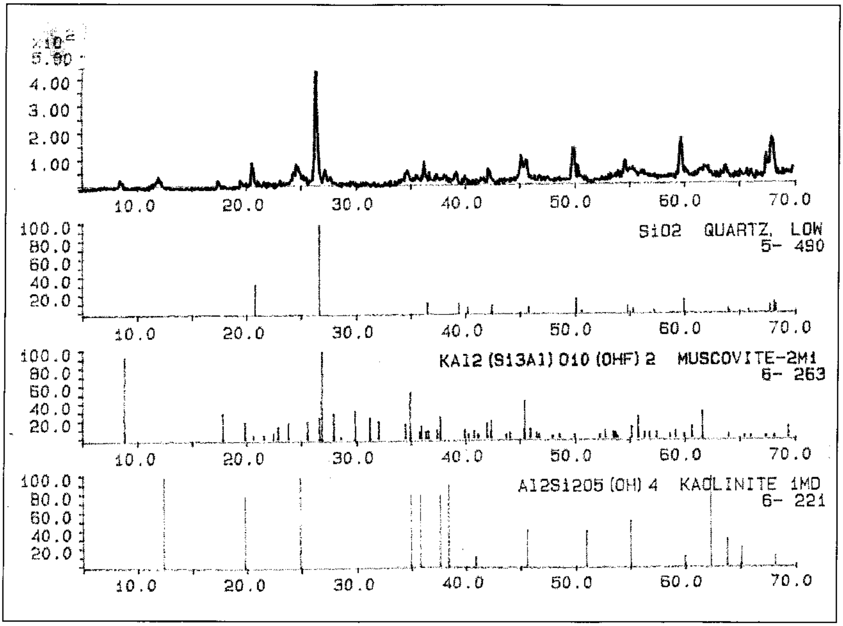


Figure 4: XRD graph analysis for clay powder sample.

## Conclusion

From this study, we found that the electroplating process is very dependent on the electroless plating process. Varying the temperature and plating time in the electroless plating process, result in different microstructures of the coating of electroplating nickel. We suggest that the optimum parameters for the electroless

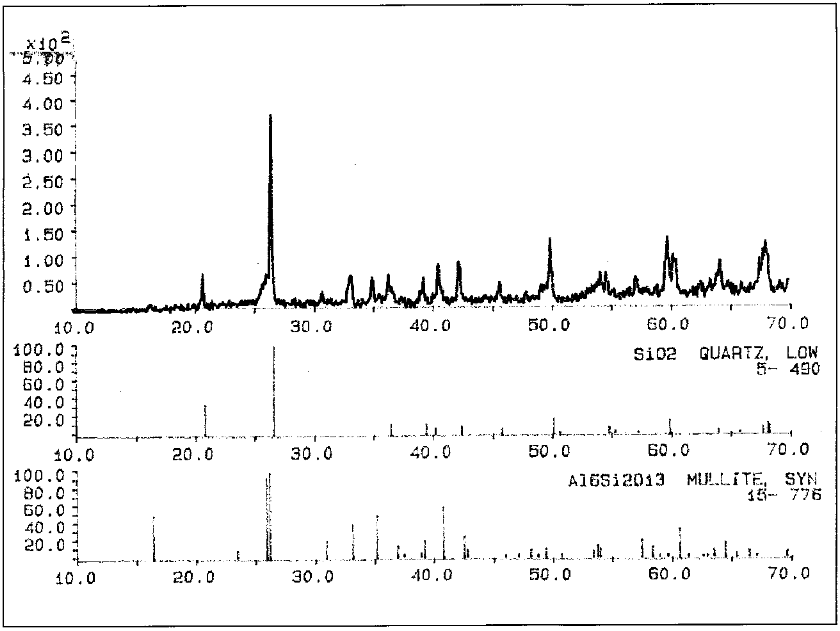


Figure 5: XRD Graph Analysis for Ceramic Plate after the Sintering Process

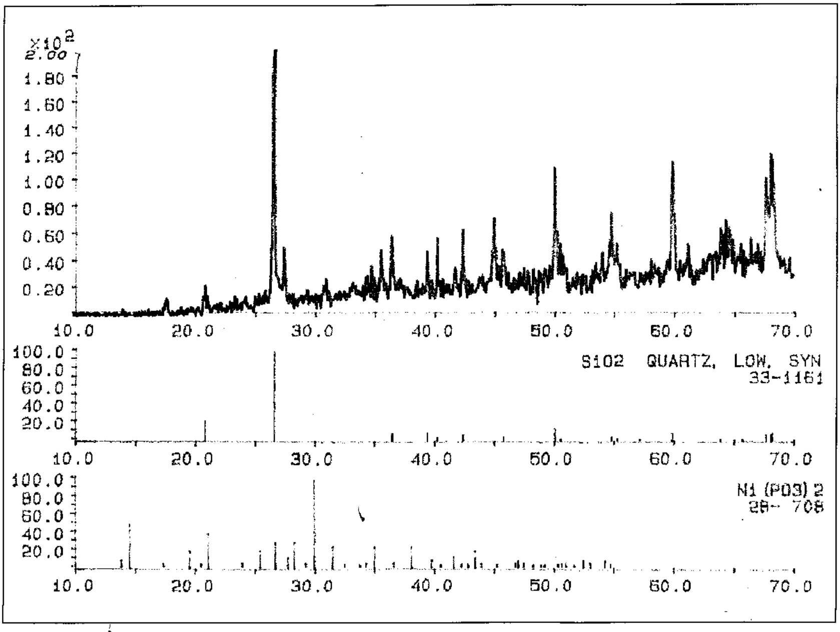


Figure 6: Xrd Graph Analysis for Ceramic Plate After the Electroless Plating Process

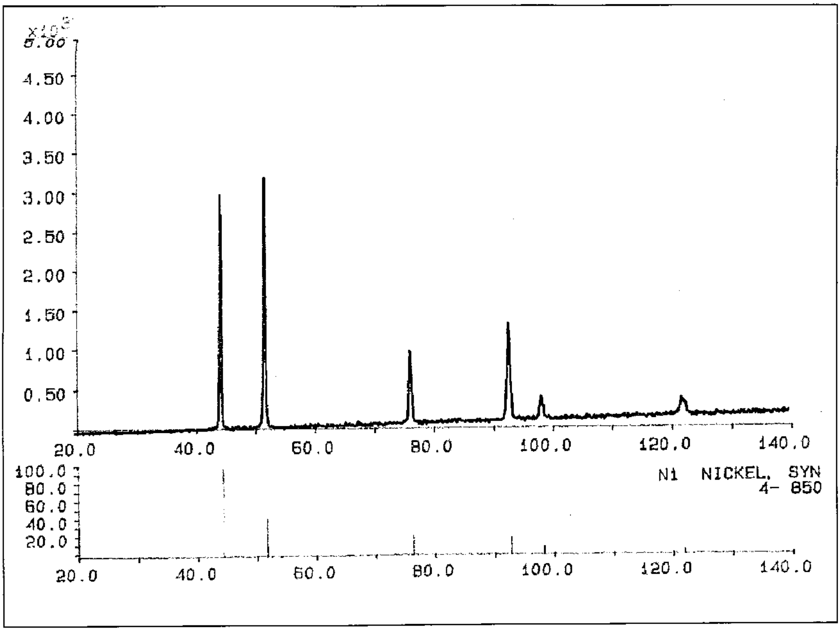


Figure 7: XRD Graph Analysis for Ceramic Plate After the Electroplating Process.

plating process for coating nickel on clay based ceramic body are at a temperature (80-95)°C and electroless plating time (15-30) minutes.

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