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# Design of Continous Set up for Synthesis of Metallic Membrane by Electroless Copper Plating

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## Abstract

The electro less plating method use in metal plating on a porous substrate is disclosed. the deposition of metal ion is done by selective contact of the plating metal salt solution with a reducing solution on the activated surface on or inside the porous substrate. Before plating process The process is accomplished by first treating with the active catalytic colloidal composition solution to provide additional active nuclei on the substrate surface, the activated substrate is then contacted with an electroless plating bath.Continuous set up is design for catalytic activity of the porous substrate by using nickel chloride as activator and for the Continous plating process using completely porous substrate with plating solution and reducing solution. The plating process in the setup is useful as it is unmanned, automatic operation for preparation of composite metallic membrane among considerably no crack or pinholes. The electro less plating method use in metal plating on a porous substrate is disclosed. After membrane form the characterization of the membrane is done by SEM and XRD.

**Keywords**: Electro less Plating, Reducing Agent, Complexing agent, Temperature, pH, Continuous Set up.

## Introduction

Electro less plating is a novel technology which is used for metallic membrane preparation. Metallic Membrane is a kind of thin layered, smooth surfaced dense material. Electro less deposition of metal on the substrate takes place resulting in the formation of metallic membrane. Various metal salts that can be reduced to metal are used in this procedure. Metallic Membranes have been widely and successfully applied in electronics, nuclear energy, chemical industry, pharmacy, foodstuff, gas separation and precision machinery for gas purification. Palladium alloy membranes are used in hydrogen separation applications.

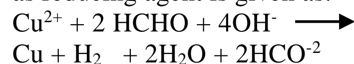
Hydrogen is released by reducing agent as hydride ion that reduces the metal salt to its metal. Several reducing agents are used in electroless plating.

Electroless Plating process is widely used for the preparation of composite metallic membrane on metal/porous substrate. However several drawbacks have been reported in literature about conventional electroless plating. When the plating metal solution and reducing solution are added together, direct contact of the solutions result in instantaneous reaction & metal is deposited in the solution rather than depositing on activated surface. This metal gets deposited in the tubing & mixing container resulting in huge wastage. This is a great loss for precious metal plating and adds to the cost of plating. Another disadvantage is that there is no assurance that all parts of substrate are plated. Also there is no assurance of complete filling, so that there are no pinholes, cracks.

## Materials and methods

The present research uses a copper salt plating solution & formaldehyde as reducing agent.

The reaction accomplished when formaldehyde is used as reducing agent is given as:

$Cu^{2+}$ + 2 HCHO + $4OH^-$ 

Cu + $H_2$ + $2H_2O$ + $2HCO^{-2}$

In this reaction, with the abundance of OH- ion, copper ion is reduced into copper atom and formaldehyde is oxidized into formic acid releasing hydrogen gas. The reaction depends on chemical concentration, temperature and pH. The catalytic activity of surface gives better deposition.

A novel electroless plating setup is described in this paper to overcome the drawbacks of conventional electroless plating process.

The electroless copper plating process is done by using continuous set up. A S.S substrate is used for plating is firstly immersed in diluted sodium hydroxide solution for 3 min to clean the surface and then into sulphuric acid for 3 min to remove of dust and also to etch the surface. The substrate is then pre-dipped into hydrochloric acid solution. Then the substrate is ready for activation step.

**Preparation of 1 liter of $NiCl_2$ & $SnCl_2$ solutions:**

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***$NiCl_2$ solution*** - 4 gm of nickel chloride is dissolved in in 200 ml of 37% of hydrochloric acid solution. Then 800 ml of deionized water is added & stirred till it is completely dissolved

***$SnCl_2$ solution*** - 8 g stannous chloride is dissolved in 200 ml of 37% of hydrochloric acid solution by stirring till it is completely dissolved. 800 ml deionized water is added later with stirring.

***TABLE1: Plating solution composition***

| | Concentration |
|---|---|
| Cupric Sulphate ($CuSO_4$ $2H_2O$) | 0.03 M |
| EDTA | 0.24 M |
| Formaldehyde (HCHO) | 0.20 M |
| 2,20-Bipyridine | 10 ppm |
| pH (NaOH) | 12.5 |
| Temperature | 65°C |

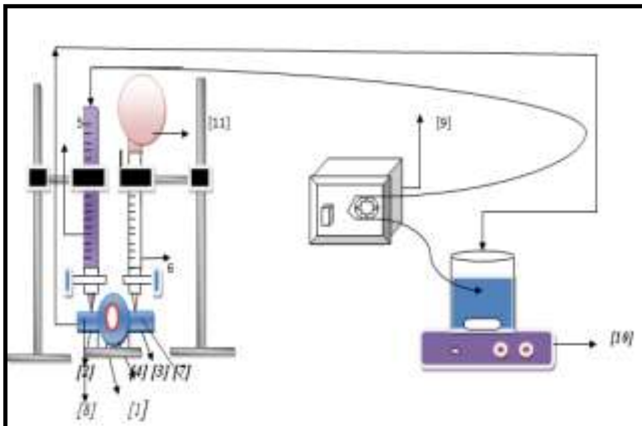

*Figure 1: Continuous Experimental Set Up*

***Set up Process:*** As per the figure 1, the set up consist of two compartment first section and second which is divided by substrate For this purpose compartment can be dismantle for removing and fitting the substrate. In first consist inlet and the second sections comprise an inlet for feed solution and an outlet for draining out the solution. Outlet is Recycle the plating solution into beaker for maintains the temperature. The inlet of the first section is connected with a first burette In second section is connected with a second burette. Flow is adjusted by valve. Valve is opened and burette filled. In embodiment of the pressurizing means is provided at the burette (1') connected with the inlet of the second section. The pressurizing means can be a balloon for desired pressure developed temperature to be maintained in the range of 55°C to 65°C. For heating purpose, the beaker is placed over the magnetic heater. It is turned on heating the solution is transfer from beaker to first section of the compartment through burette. The set up is made to be airtight and preventing leakages by sealing arrangements. Similarly, same set up is also used for sensitizing-activation of substrate. Only heating and recycling isn't required. $SnCl_2$ is subjected to pressure on one side to react with $NiCl_2$ is on activating side.

After the membrane formation is confirmed, the synthesized membrane is dryed and annealled with $N_2$ with 350ºC and leakage testing is done.

## Results and discussion

**Plating Results:** Electroless copper deposits were successfully plated in all solutions. Hydrogen bubbles could be observed during plating and the solutions remained stable and transparent in the whole process

1. Electro less plating process was prepared and it was successfully plated on or inside the substrate. So it prevents the wastage of chemicals and metal ion. And gave cost effective process.
2. Our Experimental Set up was design and gave continuous process. This was worked as cyclic manner
3. Experimental process gave complete filling of composite metallic membrane were prepared which is hydrogen permeable pure membrane. And there was no air leakage and no pin holes or Crack observed.

**SEM Analysis (scanning electron microscopy):** The Surface morphology test were carried out by SEM analysis.Which give continous and smooth deposition of copper metal ion on and inside the substrate. The result of SEM analysis are shown in figure 2.

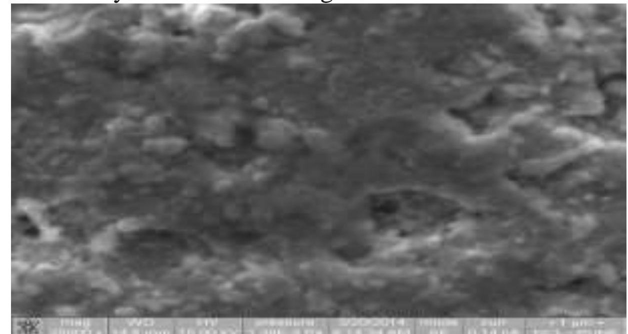

*Figure 2 : SEM for Electroless copper deposite(mag25,000 X)*

**XRD analysis (X-ray diffraction):** The crystal structure and surface residual stress test were carried out by XRD unit. The crystalline peaks in figure are resulted from the Cu substrate. A broad peak appearing around 2Ø of 829

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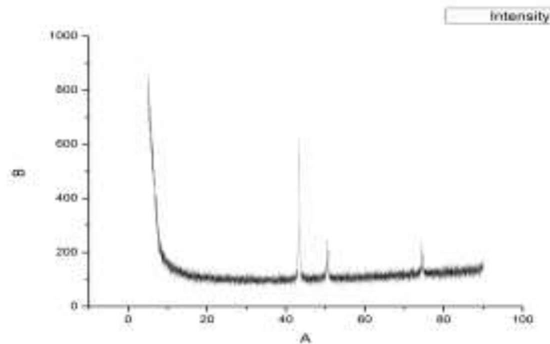
indicates the X-ray diffraction.The Result of XRD are shown in below figure 3.

*XRD Analysis: (X- ray Diffraction Studies)*




***On X axis : A - 2 (Theta) degree.***
***On Y axis : B – Intensity.***
***Figure 3.XRD analysis of Electroless Copper coatings (Annealed at 400ºc for 1 hr.)***

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## Conclusion

Electro less plating of Cu was done on the substrate with formaldehyde as reducing agent. No metal deposition was observed in the tube & containers. Our Experimental Set up was gave continuous plating. The surface morphology and residual stress of the deposits were examined using SEM and XRD methods.

The results show that: Deposits gives Continous and smoother surface deposition can be obtained by using copper as metal ion and formaldehyde as reducing agent in the electroless copper plating bath operated at 65°C.and there is no significant amount of residual stress on the substrate surface of the deposits.

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## Author Biblography

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