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# Porosity study on microstructures of nickel-chromium alloy plating on copper substrate

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**Abstract:** *Porosity in coatings is a vital parameter for many engineering applications. Having a porous coating is good with respect to few aspects and bad with respect to others. One of the main applications of porous metal coating is in the field of membrane filters for oil water separation to deal with industrial waste water and off-shore oil spill. It is also used for electrolytic cleaning (purification) of water. Studying the nature of formation and development of pores is being carried out by many researchers to determine the parameters affecting the porosity of coatings. Optimizing the parameters to obtain a favourable coating with least defect is the idea behind these research topics. Nickel and chromium coatings are widely used for many industrial applications because of specific properties. Nickel-chromium alloy coatings offer better hardness, wear and corrosion resistance compared to individual nickel or chromium coatings. The alloy coatings are observed to be porous in nature compared to individual nickel and chromium coatings. Controlling the porosity of the deposition by varying the process parameters is a very interesting topic of research as it greatly influences the properties. In the present work, the coating microstructures of nickel-chromium alloys developed by electrodeposition techniques are analysed and evaluated using Field Emission Scanning Electron Microscopy (FESEM) for varying current densities and plating time resulting a good uniform coating with varying porosity.*

**Keywords:** Nickel; Chromium; Electrodeposition; Filter membranes; Electrolytic cleaning.

## 1. Introduction

Electroplating which was earlier used for decoration purpose is presently used for many advanced applications imparting corrosion and wear resistance. It is also used for many electronic applications [1]. Nickel-chromium alloys, commonly known as nichrome alloys are promising material for different industrial applications because of their excellent wear and scratch resistant properties. Nichrome materials also find application in various high temperature environments as they are more resistant to oxidation compared to nickel or chromium [2]. Coating by electrodeposition is a major effective method for developing nanocrystalline coatings. The nanocoatings produced by electro-deposition would be uniform and produced in bulk. The initial investment required for electrodeposition would be less compared to other deposition techniques [3]. There were many serious problems associated with traditional chromium plating based on chromic acid. It is because of this reason that chromium is coated along with some other alloying element thereby reducing the problems associated with industrial chromium plating [4]. In addition, it is very much essential to replace hexavalent chromium electrolyte with trivalent chromium electrolyte as it is toxic and pose a lot of environmental effects. However trivalent chromium electrolytes tried are yet to reach an optimum condition [5]. It is due to these shortcomings that chromium is normally alloyed with some other elements to get optimum properties of coats. In recent times electrodeposition of iron group metals and their alloys is considered for their unique magnetic and thermophysical properties [6]. From the literatures it is observed that nickel-chromium alloy coatings are normally deposited onto copper, steel, aluminum and stainless-steel substrates. The deposition techniques normally employed to deposit Ni-Cr alloy is electrodeposition and thermal spray. Nickel-chromium alloy coatings find application in many industries because of its unique hardness, wear and corrosion resistant properties. Also, the coatings are observed to be porous in nature imparting unique properties. From the literatures it is observed that several process parameters such as current density, gas bubbles, feeding mechanism, electrolyte composition, plating time and pulse parameters influence the quality of the electrodeposited part in terms of its porosity [7-11].

Many studies are underway on effective oil-water separation techniques for pollution control and oil spill recovery. In recent times filter membranes are being studied with great interest and are made of metals, polymers and fibers (Fig.1). Filter membranes need to be of high porosity and of opposite wettability to aqueous

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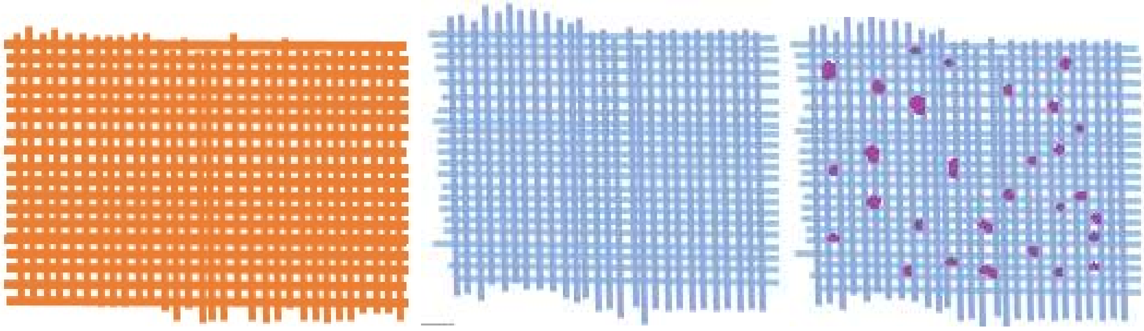
and oil phase. Another application of porous coatings is in electrolytic purification of water wherein the porous coated metal is used as cathode in the electrolytic cell and the heavier impure atoms in the solution are deposited onto the porous cathode metal [12]. In the present study nickel-chromium alloy coatings are deposited onto copper substrate by electrodeposition technique for varying current density and plating time conditions. The coatings were studied for their porous nature using Field Emission Scanning Electron Microscope (FESEM) so that it can be used for filter membrane application or as an electrode material for electrolytic purification of water.



(a) (b) (c)

***Fig.1**. Representation of a) Filter membrane made of copper b) Ni-Cr alloy coated membrane and c) oil particulates entangled in filter membrane.*

## 2. Experimental

Nickel-chromium alloy coatings were developed on copper substrate by electrodeposition route. Before the surface was coated it was polished to mirror like finish using grit sheets. The polished surfaces were first given a thin layer coating of nickel by watts bright electroplating bath solution. This thin layer of coating gives a good bonding to nickel-chromium alloy deposits. The plating bath composition used for the alloy deposition includes chromium chloride ($CrCl_3.6H_2O$) 40 g/L, nickel chloride ($NiCl_2.6H_2O$) 200 g/L, and boric acid ($H_3BO_3$) 40 g/L. pH of the bath was maintained at 3. Air agitation technique was used to mix the bath mixture during the plating process. Lead strip was taken as anode and the copper substrate on which coating is performed is taken as cathode. To study the porosity effect on coatings developed, the current density and plating time was varied and the effect of this variation on porosity was analyzed using FESEM. Current density variation was studied in the range of 2-8 A/dm$^2$ in steps of 2 A/dm$^2$ and plating time was varied between 5 and 25 min in steps of 5 min by keeping current density constant. The plating electrolyte composition and plating parameters studied are represented in Table 1 and Table 2.

***Table 1***
*Plating parameters for varying current density in nickel-chromium plating at constant plating time.*

| Parameter | Condition |
|---|---|
| Bath temperature | 300C |
| Type of agitation | Air |
| Anode | Lead |
| Cathode | Copper strip |
| pH | 3 |
| Current density | 2-8 A/dm$^2$ |
| Plating time | 10 min |

***Table 2***
*Plating parameters for varying current density in nickel-chromium plating at constant current density.*

| Parameter | Condition |
|---|---|
| Bath temperature | 30 °C |
| Type of agitation | Air |
| Anode | Lead |
| Cathode | Copper strip |
| pH | 3 |
| Current density | 10 A/dm$^2$ |
| Plating time | 10-25 min |

## 3. Results and discussion

### *3.1. Analysis of porosity of coatings*

Microstructures of coated samples were captured using optical image analyzer to study if the coatings are porous in nature. Fig.2 shows the microstructures captured for Ni-Cr alloy coating at 8 A/dm$^2$ current density and plating time of 10 min at 50X and 100X magnifications. The coatings are observed to be porous. To study in detail about the size of pores, number of pores and its variation with respect to plating time and current densities the samples were analyzed under Field Emission Scanning Electron Microscope (FESEM).

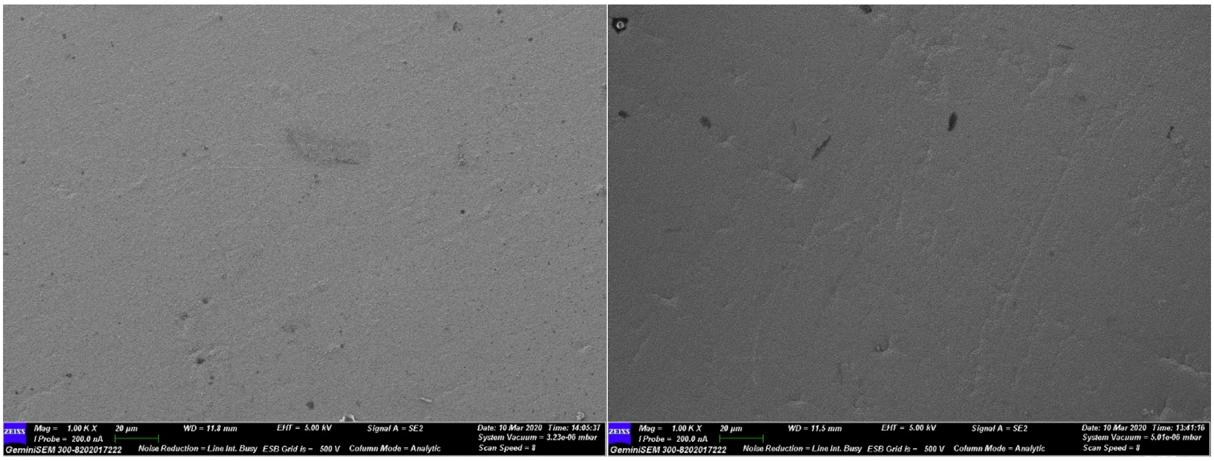
The microstructures of coating of nickel-chromium alloy were captured during the deposition process to understand the microstructural cause of porosity which is very critical in determining the properties of coating. Porosity in alloy plating is represented by the packing of nickel and chromium crystals from the nucleation sites. Porosity of coating is studied to have an impact on the inherent properties of the component. Porosity of coating is determined in terms of pore size distribution, relative frequency and average pore size in the coating produced.

### *3.2. Effect of current density on coating microstructures*

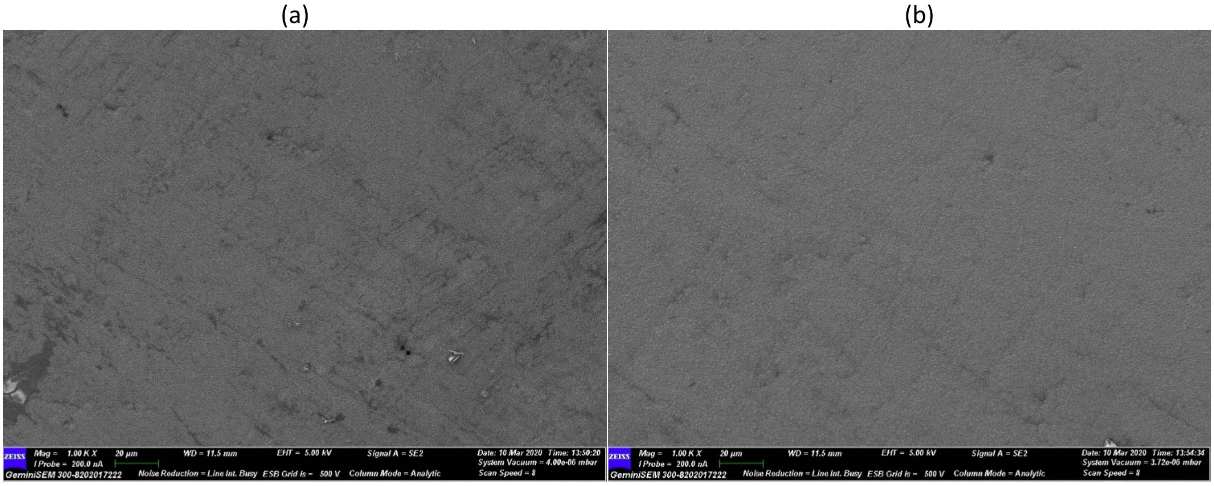
FESEM images of deposits of nickel-chromium alloy were studied for nickel and chromium crystals. Fig.3 shows the microstructures of nickel-chromium alloy coatings deposited onto copper by electrodeposition process under varying current density from 2 to 8 A/dm$^2$ in steps of 2 A/dm$^2$. The deposits in this current density range were observed to be of very smooth surface with smaller grain size. From the microstructures it can be seen that as the current density of coating rises from 2 A/dm$^2$ to 8 A/dm$^2$, the porosity i.e the area occupied by pores during the coating increases in the coating. There are many reasons for this phenomenon as studied from literatures. The rise in porosity of coating with rise in current density is due two main reasons as observed from literatures [13];

i) Depletion of ions leading to bubble formation at higher current densities.
ii) Emission of hydrogen bubbles will reduce for lower current densities, leading to improved quality of deposit.


***Fig.2***. *Microstructures of Ni-Cr alloy plated sample at 8 A/dm$^2$ current density and plating time of 10 min for 50X and 100X magnification.*



(a) (b)



(c) (d)

***Fig.3***. *Ni-Cr alloy coating microstructures for varying plating current densities: a) 2 A/dm$^2$, b) 4 A/dm$^2$, c) 6 A/dm$^2$ and d) 8 A/dm$^2$*

The porosity of coatings can be studied through the number of pores and size of pores in the microstructure. From Fig.4, It can be observed that the number of pores reduce with rise in current density but the size of pores increases with current density indicating a larger area of porous coating. The pore size was reduced drastically for current density 8 A/dm$^2$ as compared to microstructures of 4 and 6 A/dm$^2$. This observation is due to the formation of secondary nickel nodules at higher current densities. The secondary nodules formed will cover the pores thereby reducing the pore size and pores area. From the study in the current density range of 2–8 A/dm$^2$, it is seen that the pores are uniformly distributed, smaller in size and very large in number at low current density of 2 A/dm$^2$. This type of uniformly distributed small sized pores is very much suitable for filter membranes in oil water separation. Whereas the other type of porous coating observed for higher current densities wherein the size of pores is large and less in number is suitable for electrolytic purification of water. However, it is observed from the microstructures that the size of pores was reduced at high current density of 8 A/dm$^2$ due to the formation of secondary nodules which cover the porous films. The secondary nickel nodules are larger in size compared to primary nickel nodules

### *3.3. Effect of plating time on coating microstructures*

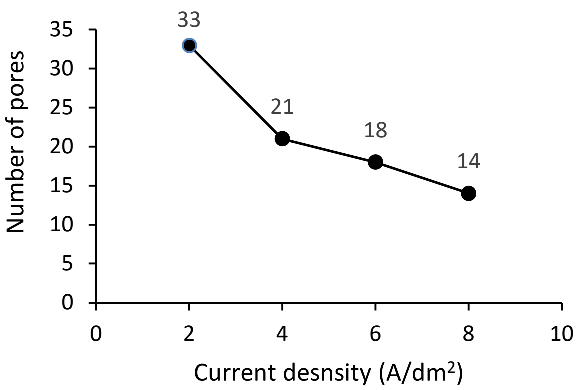
Deposits of nickel-chromium alloy for varying plating time by keeping current density constant at 10 A/dm$^2$ is studied for porosity. Microstructures captured by FESEM for plating time in the range of 10-25 min in steps of 5 min at 1000 X magnification shows a good representation of porosity of coatings (Fig.5). For a coating time of 10 min the coating is observed to be patchy and non-uniform. The size of the pores being large and less in number. The coatings are observed to be uniformly porous for coating time of 15 min. As the plating time is increased further the size of pores reduce and number increases forming a good uniformly porous coating over the substrate.

It is observed from the captured microstructures that the number of pores increase with increase in plating time but the size of pores goes on reducing. This indicates that the coatings produced at low plating time is more suitable for electrolytic purification of water and coatings produced at high plating time is applicable for filter membranes. But for plating time of 25 min it is observed that the number of pores reduce drastically and the size of pores increase in the coating. This rise in porosity for very high plating times is attributed to the fact that ions deplete for higher plating times leading to hydrogen release and bubble formation thereby increasing the size of pores [14].

From the microstructural study on the porosity of coating it can be generalized that nickel-chromium alloy coatings produced are porous in nature. The nature of porosity variation with respect to current density and plating time indicates some porous coatings are more suitable for filter membrane application and others are suitable for electrolytic water purification application as can be deduced from Table 3.

***Table 3***
*Application of nickel-chromium plating based on plating parameters (current density/plating time)*

| Plating parameters | Low | High |
|---|---|---|
| Current density | Filter membranes | Electrolytic Purification |
| Plating time | Electrolytic Purification | Filter membranes |



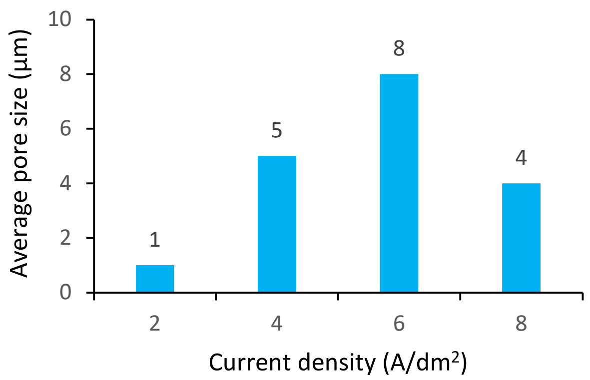




***Fig.4**. Variation in number of pores and Size of the largest pore with current density*

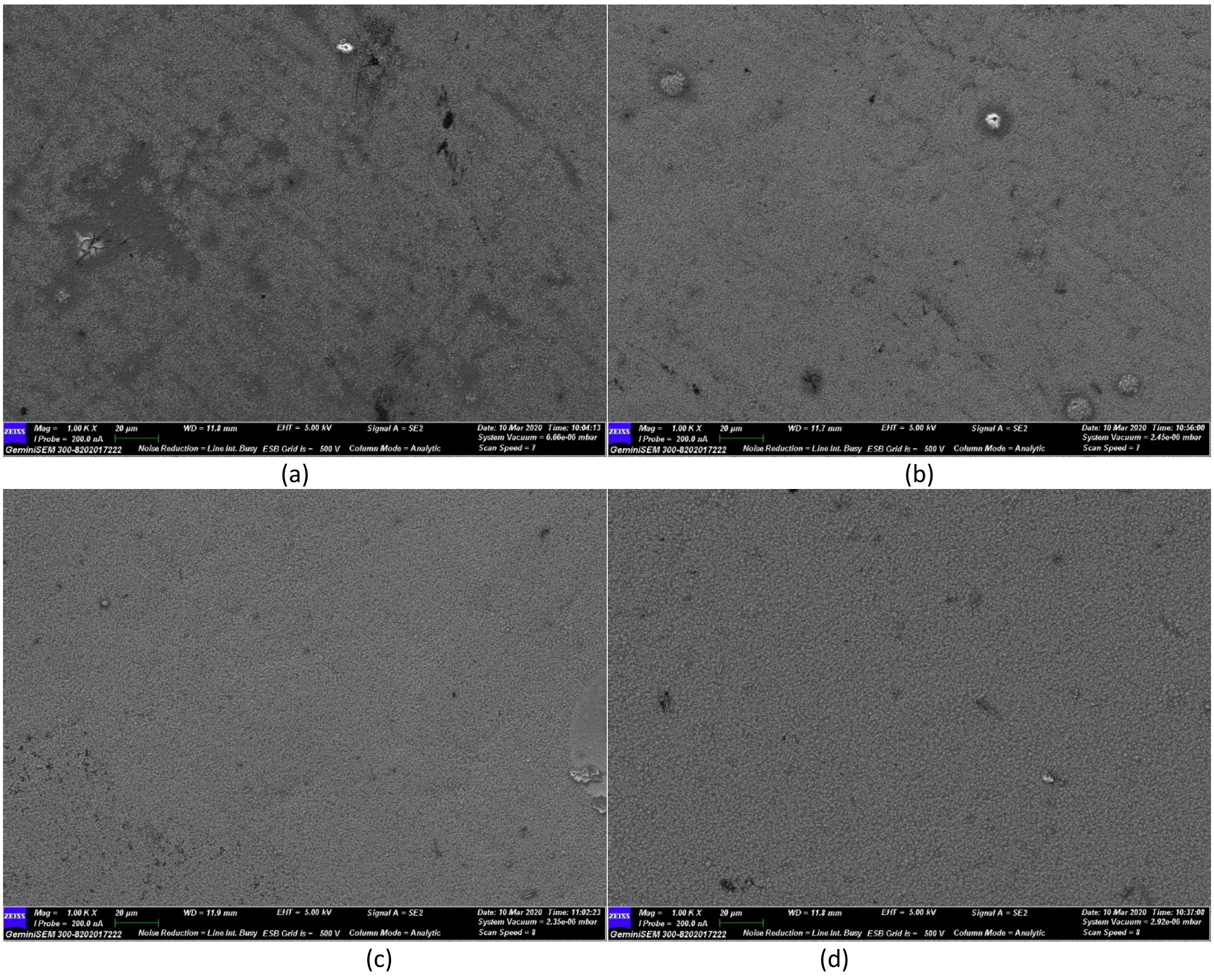



***Fig.5**. Ni-Cr alloy coating microstructures for varying plating time at a constant current density of 10 A/dm$^2$. a) 10 min, b) 15 min, c) 20 min and d) 25 min.*

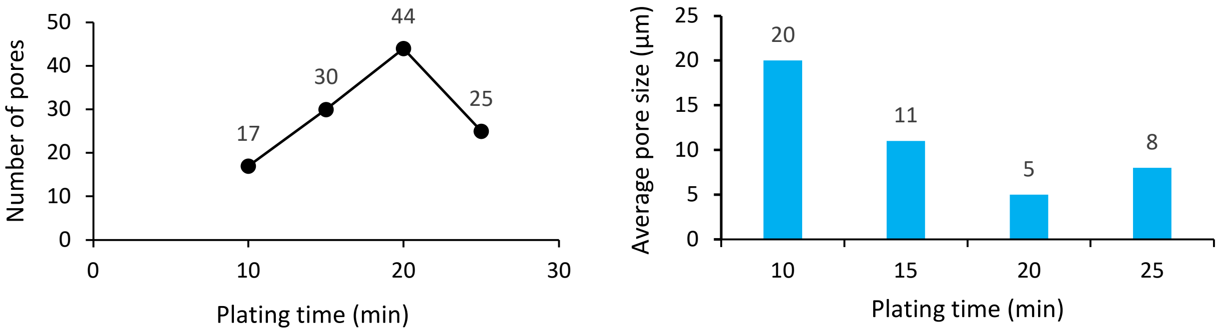



***Fig.6**. Variation in number of pores and Size of the largest pore with plating time*

## 4. Conclusion

The following observations were recorded after the Ni-Cr alloy coatings were deposited onto copper substrate:

i) The coatings were observed to be porous in nature with very small grain particles of nickel and chromium distributed uniformly.

ii) The porous nature of alloy coatings finds its application in the field of filter membranes and electrolytic purification.

iii) At low current densities the pores observed in coatings are very small in size and large in number and uniformly distributed. At high current densities the pores are of larger size and small in number.

iv) For low plating time the pores obtained in coatings are of large size and small in number whereas for higher plating times small sized uniformly distributed pores are produced.

v) Small sized uniformly spaced porous coating is more suitable for filter membranes and large sized porous coatings are more suitable for electrode material in electrolytic purification of water.

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