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Effect of Current Density And Heat Treatment on The Electrodeposited Nickel Coated Bulk Graphite

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Abstract

In this work , a bulk graphite substrate was coated with nickel using electroplating technology. The reason of this work is to remove the oxidation characteristics of graphite at intermediate temperatures in the range 450– 500 °C. The experimental facilities were locally designed and developed . The visualization of the coating topology showed the effect of current density on the microstructure of the electrodeposited layers. The grain size tends to be smaller as the current densities are increased . The effect of heat treatment on the microstructure showed an encouraged grain growth which was greater at high treatment temperature .

تأثير كثافة تيار الترسيب و المعاملة الحرارية على النيكل المرسبة على الكرافيت

الخلاصة

في هذا البحث تم ترسيب طبقة نيكل على نموذج من الكرافيت باستخدام تقنية الطلاء الكهربائي.ان الهدف من الدراسة إزالة صفة التأكسد للكرافيت في درجات الحرارة المتوسطة للمدى (450 -500 ℃.) . تم تصميم و تطوير الوسائل التجريبية محليا. أوضحت دراسة طبوغرافية سطح الطلاءات تأثير كثافة تيار الترسيب والمعاملة الحرارية على التركيب الداخلي لطبقات الطلاء.ان نتائج التركيب الدقيق أظهرت إن الحجم الحبيبي لطبقات الطلاء تميل لان تصبح اصغر مع زيادة كثافة تيار الترسيب . أما نتائج تأثير المعاملة الحرارية فقد أظهرت حدوث نمو حبيبي و يزداد مع زيادة درجة حرارة المعاملة .

Introduction

Electroplating is the application of a metal coating to a metallic or other conducting surface by an electrochemical process. The article to be plated (the work) is made as cathode (negative electrode) of an electrolysiscell through which a direct electric current is passed. The article is immersed in an aqueous solution (the bath) containing the required metal in an oxidized form, either as an equated cation or as a complex ion. The anode is usually a bar of the metal of plating. During electrolysis, metal is deposited on the work, and metal from the bar dissolves [1, 2]:

$$M^{z+} + ze \rightleftharpoons M \qquad ...... (1)$$

Some of the purposes for which articles are electroplated [3]:

1-Appearance.

2-Protection of oxidization.

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3-Special surface properties (smooth, shine and hard).

4-Engineering or mechanical properties.

This process can be represented as in figure 1 [1].

Faraday's laws of electrolysis govern the amount of metal deposited.

$$w = ZQ = \frac{A_{wt}}{nF} Q \qquad \text{..... (2)}$$

Where A_{wt} – is the atomic weight of metal deposited on the cathode,

n –Number of electrons involved in the deposition reaction,

Q- is the product of the current I ,and the time t,

Z- is the electrochemical equivalent ,the constant of proportionality , and F=96.487 $C.mol^{-1.}$

It is known that the microstructure and mechanical properties of a plated film depend upon the plating conditions such as temperature, PH, current density, etc [4].

Crystallographically perfect graphite is a rigid material that is resistance to stretching and compression, a black, lustrous solid .It has a density of 2.265 g/ml, it is a good conductor of heat and electricity, and is chemically inert. Thus, it oxidizes only slowly above 600 °C in air, and is attacked only slowly, by most chemical reagents [5]. In this work, we report the results of an experimental investigation into the effect of current density on the microstructure of electrodeposited nickel on bulk graphite substrate According to Faraday's law, the plating rate is proportional to the current density according to the equation .

$$v = CE \frac{i_c}{zF} \left[{mol}/{(cm^2 s)} \right] \qquad \text{.... (3)}$$

Where CE - is the current efficiency and i_c - is cathodic current density.

Experimental procedure

Graphite substrates were prepared by cutting the graphite rods to the form of disc with diameter about 3 cm and thickness about 1 cm. The graphite disc had a very rough and irregular surface. So it must be removed by machining using grinding with water , but the surface should have a little degree of roughness so as to allow the coating to be bonded to the substrate . The machining was carried out on each side for equal thickness. To obtain a clean surface, the following cleaning steps were used , alkaline cleaning and pickling .

Nickel coatings were electroplated from a Watt's solution, consisting of the salts listed in table 1 [4].

The electro deposition was carried out in a glass container (Pyrex) with a capacity of (5 liter) with dimensions (36 cm × 15 cm × 20cm).A heating element connected to a temperature controller was used to control the required temperature of the bath . To apply the current required , power supply was used with range of (0-15 Amp.) and voltage (10 volt). And to insurance the homogeneity of the temperature and concentration of the solution, a water pump was used .

To measure the PH of the solution , PH meter of type (Bibby) was used in this process. Rod of nickel with purity 99.9% was used as anode material . The solution is heated to the desired value ,using a temperature controller is capable of achieving a temperature stability of ±1 . After this, a direct current was applied of different values. Before going into microstructure test , the surface of specimens was etched with (50 mol HNO_3 and 50 ml acetic acid) , mixing fresh , immerse for 10 sec according to the ASTM 407 standard [6].

Results and Discussion

The microstructure of electrodeposited nickel was found to be very sensitive to variations in the electrodepositing conditions. They are found to basically affect the grain size , which in turn affect the microstructure and mechanical properties . After preparing the substrates, mentioned above, the cleaned surfaces were prepared for optical microscopy analysis before coating with electrodeposited nickel .Figure 2 shows the bulk graphite surface before coating .

When the current passes through electrolyte at optimal temperature of 60 °C, and because of the good conductivity of graphite substrate this creates a high potential difference at the surface of cathode, at which the positive ions discharged cause an increase in the mobility of hydrogen and nickel ions.

Figure 3 shows the effect of current density on the microstructure of the deposited layer.

From figure 3 a and b , it is noticed that at low current density the grain size of deposited particles is increased due to the low rate of deposition and the deposited nickel has rather lower throwing power toward the cathode . This allows nickel atoms to occupy suitable position within the previously deposited atoms at the substrate , and consequently nickel grows to form large grain . As the current density is increased , figure 3 c , the rate of deposition is increased and so dose the throwing power . So the increase in the deposition rate may leads to increase in nucleation rates , resulting in small grains . The deposited nickel is hence dense and strong.

At high current density, the plating rate is high. Atoms may pile up with in sufficient time to relax , and hence leave behind pores with relatively weak bonding between particles in the deposited layer . The coating is porous, with a fine grained structure, as shown in figure 3d. The variation in the thickness of the deposited nickel with current density as shown in figure 4.Figure 5 shows the effects of heat treatment temperatures on the microstructure of coated layers at different temp. for one hour Figure

5 a shows the coating layer before heat treatment process . It is noticed that grain size of the deposited particles is small with presence of pitting . Pitting is caused by many factors including adhesion of air or hydrogen bubbles that can result from a solution that is chemically out of balance , as too low PH , or is inadequately agitated. After heat treatment at 200 °C for one hour ,we can see that decrease in pitting with a simple increase in grain size of deposited layer and a limited crystal growth ,as shown in figure 5 b . When heat treatment temperature is raised to 220 °C and 240°C,figure 5 c and d respectively , the grain size of electrodeposited nickel is found to be increased with increasing heat treatment temperatures. These grains were grown on the expense of the smaller ones. At heat treatment temperature as high as 240°C,a further significant grain growth occurred, with the grains tending to change their overall shapes leading to change the properties of coatings .

Conclusions

Based on experimental result presented in this work, it is possible to draw the following conclusions:

1-The deposition parameters exhibited a strong and direct relation to the final microstructure of the deposits .

2-The variation of deposition conditions such as current density affect obtaining a high quality electrodeposited layer .

3-Determenation and controlling on the annealing process and its effect on the nature of the high quality deposited layer.

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Table (1) Shows the solution contraction.

Materials	Symbol	Concentration
Nickel sulfate	$NiSO_4.6H_2O$	270 g/l
Nickel chloride	$NiCl_2.6H_2O$	40 g/l
Boric acid	HBO_3	30 g/l
Brighteners	Organic material	2 ml/l

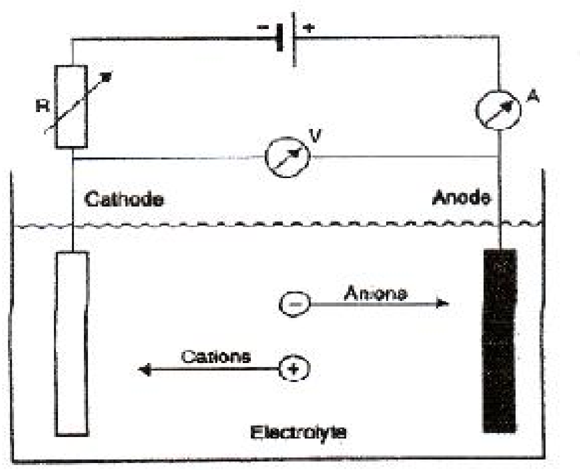

Figure (1) Main components of a DC electrolysis system.

Figure (2) The bulk graphite surface before coating.

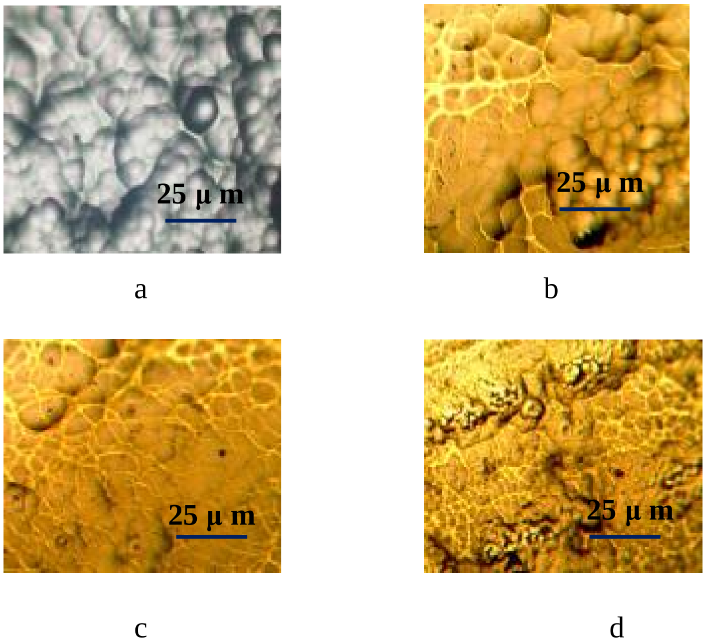

Figure (3) Effect of current density on the microstructure of the deposited layer. (J=3.3, 4.8, 6.5 and 8.2 A /dm ² respectively, D = 12 cm, t = 25 min and PH= 3.5- 4

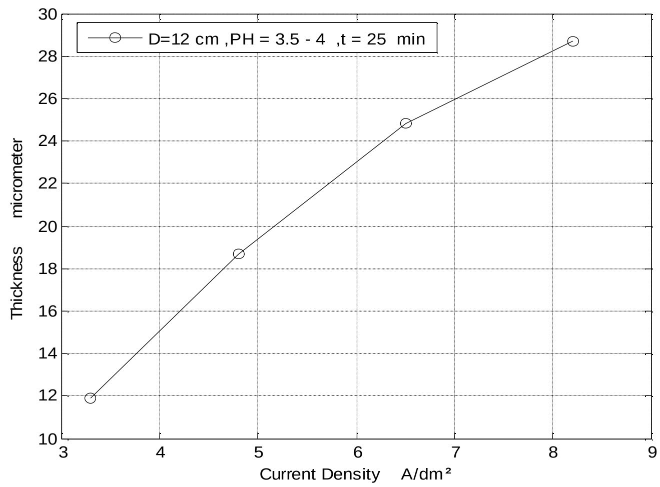

Figure (4) Thickness variation of electrodeposited nickel with current density

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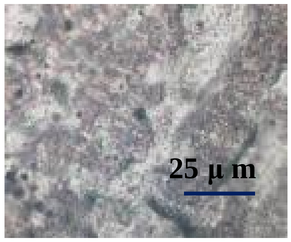

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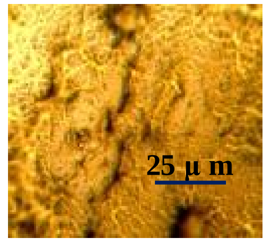

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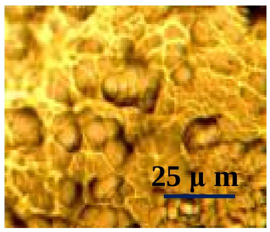

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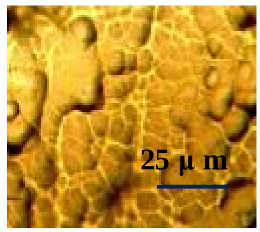

d

Figure (5) Effects of heat treatment temperatures on microstructure of coating layers (T_a = 200 , 220 and 240 °C respectively , J = 6.5 A /dm ^{2} , D = 12 cm , t = 25 min , PH = 3.5 – 4)