Electrodeposition and characterization of nano-structured black nickel thin films

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Abstract: The electrodeposition and characterization of nano-structured black nickel coatings were presented. The influences of bath pH, electrodeposition time, stirring speed, temperature and current density on the color and microstructure of the electrodeposited nickel film were investigated through naked eyes, scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques. Meanwhile, the corrosion resistance of the optimized black nickel film was evaluated by the polarization measurement and electrochemical impedance spectroscopy (EIS) in the neutral 3.5% NaCl solution. The results show that the color of the electrodeposited nickel film was highly dependent on the above technological parameters. The operating parameters were optimized mainly according to the color. The optimized black nickel film possesses nano-structure with an average grain diameter of about 50 nm. It also exhibits enhanced corrosion resistance when compared with white nickel coatings electrdodeposited under the same condition except the variation of the electroplating current density.

Key words: electrodeposition; nano-black nickel film; corrosion

1 Introduction

Thin black nickel layers are widely used as decorative and protective films for iron-based substratum, and they are also candidates as active solar-thermal coatings for IR absorbers [1,2]. Thin black nickel films have been developed for over 50 years [3]. Nowadays, different techniques such as chemical conversation, thermal oxidation of metallic films [4], sputtering [5], spray pyrolysis deposition [6] and electroless and electrodeposition [4,7,8] have been reported.

Electrodeposition possesses many advantages including low cost and industrial applicability, simple operation, versatility, few size and shape limitations, and high probability of transferring this technology to existing electrodeposition and electroforming industries [9]. The preparation of black coatings by electro-deposition technique has therefore aroused great attention in the world [10−13]. The electrodeposition of nickel-based black coatings was reported [10,11]. Unlike

black chromium [11,12] or black cobalt [14] coatings, it is possible to produce a variety of nickel-based black coatings containing different elements such as zinc, cadmium, molybdenum and tin [15].

On the other hand, preparation and characterization of nano-structured films are important and active research objectives in the materials science field for a long time. This interest comes mostly from the unique chemical, physical, optical and mechanical properties of nano-structured materials [9]. The nano-structured materials possess a large number of grain boundaries, of which the volume is close to 50% of the total volume of the material and are the origin of their unusual properties.

In this work, nano-structured black nickel film was electrodeposited on brass copper substrate (64Cu36Zn) using traditional DC electrodeposition technique, and the influences of principal technological parameters such as agitation speed, current density, bath pH and temperature on the color and morphology of the electrodeposited nickel film were investigated. The structure and morphology of the optimized nano-structured black

nickel film were characterized using SEM and XRD. Its corrosion resistance was studied using polarization and electrochemical impedance spectroscopy (EIS).

2 Experimental

2.1 Preparation of nano-structured black nickel coating

The electrodeposition was performed in a double-electrode cell. A large bright platinum foil was used as anode. Brass copper (64Cu36Zn) cylinder with an exposed area of 0.5 cm^2 was used as cathode, and positioned in vertical plane with the anode. The distance between the cathode and anode was 1 cm.

The electrodeposition baths were prepared using AR grade reagents and twice distilled water. The compositions and operating parameters for electro-deposition are shown in Table 1. In order to study the influence of operating parameters, the bath temperature, bath pH, current density (J), stirring speed and electrodeposition time (t) are listed in Table 1.

Prior to electrodeposition, the substrates were sequentially polished using silicon carbide emery papers and velvet, rinsed in twice-distilled water and acetone, and then blow-dried by air.

The electrodeposited nickel film color was observed by naked eyes. The surface morphologies were investigated using a scanning electron microscope (SEM, HIAACHIS−4700). Its crystal structure was evaluated by X-ray diffraction (XRD, D8 Advance Bruker) with Cu K$_\alpha$ radiation at a scan rate of 4 (°)/min in a scanning range of 30°−90°.

2.2 Corrosion characterization of nano-structured black nickel thin film

According to the study of influence of operating parameters, optimized operating parameters were chosen. Then, the corrosion behavior of the optimized black thin nickel film was evaluated through polarization measurement and EIS. The electrochemical measurements were performed in a three-electrode cell in 3.5% NaCl solution (pH=6.87) at 25 °C. A saturated calomel electrode (SCE) and a large bright platinum foil were used as reference and auxiliary electrodes, respectively.

The polarization measurements were performed using an electrochemical workstation (CHI660A, China).

The polarization curves were recorded by sweeping the potential between −0.69 V and 1.20 V vs open circuit potential (OCP) at a scan rate of 0.001 V/s. The EIS measurements were conducted in the frequency range from 60 kHz to 0.01 Hz with 10 mV sinusoidal signal amplitude using an impedance measurement unit (IM 6e, Australia). Each experiment was repeated at least three times to verify the reproducibility of the results. For comparison, white nickel film was also electrodeposited using the same technological parameters except the current density, its polarization and EIS were then studied.

3 Results and discussion

3.1 Influence of technological parameters on nickel film

In our previous electrodeposition research [16,17], it was found that the morphology and structure of the electrodeposited nickel films depended mostly on the electrodeposition solution pH, current density, stirring speed and the bath temperature. Consequently, the influences of the above principal technological parameters were investigated. The purpose was to prepare black nickel film. The color of the film was therefore concentrated.

The pH of the electrodeposition solution is of key importance, because it determines the color of the electrodeposited thin nickel film. The electrodeposition solution pH varied between 2.0 and 6.0 and was adjusted using sulphuric acid with a concentration of 10%. The color of the film electrodeposited at different pH is shown in Table 2. Here, the temperature, current density, electrodeposition time and stirring speed were fixed as 60 °C, 3.0 mA/cm^2, 30 min, 900 r/min, respectively. At lower pH such as 2.0−3.0, the film displayed black color. When the pH was raised to 4.0, gray films were electrodeposited. At higher pH between 5.0 and 6.0, the electrodeposited film showed white color. For the reason that the lower the solution pH, the more intense H$_2$ evolution which is always coupled with nickel electrodeposition, the pH for black nickel film electrodeposition was hereinafter fixed as 3.0. Figure 1 shows the surface morphology of the Ni film electrodeposited at pH 3. The spherical nickel grains are uniformly distributed, and the nickel film possesses nanostructure with a grain diameter less than 100 nm.

Table 1 Composition of electrolyte and deposition parameters

Composition/(g·L^{-1})			Additives	pH	Temperature/ °C	Current density/ (mA·cm^{-2})	Stirring speed/ (r·min^{-1})	Electrodeposition time/min
NiSO$_4$·6H$_2$O	NiCl$_2$·6H$_2$O	H$_3$BO$_3$						
100	40	30	A little	2.0−6.0	20−80	1−5	600−1200	10−60

Table 2 Color of nickel film electrodeposited at different pH (θ=60 °C, J=3.0 mA/cm^2, t=30 min, stirring speed =900 r/min)

pH	2.0−3.0	4.0	5.0−6.0
Color	Black	Gray	White
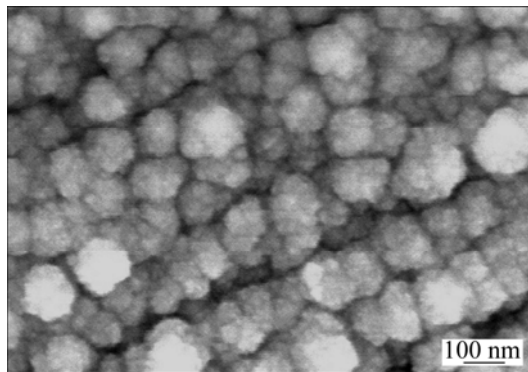
Fig. 1 SEM image of nickel film electrodeposited at pH 3.0 (θ= 60 °C, J=3.0 mA/cm^2, t=30 min, stirring speed =900 r/min)

As other technological parameters were fixed as θ=60 °C, pH=3.0, t=30 min, stirring speed = 900 r/min, the color of the film electrodeposited at different current densities is shown in Table 3. At lower current density of 1.0−3.0 mA/cm^2, the nickel film displays black color. At 4.0 mA/cm^2, it shows gray color. As current density was further increased to 5.0 mA/cm^2, just bright nickel coating was electrodeposited. Hence, the appropriate current density for black Ni film electrodeposition was chosen in the range of 1.0−3.0 mA/cm^2. Surface morphologies of the film electrodeposited at current densities of 1.0−3.0 mA/cm^2 are shown in Fig. 2. As the current density increases, the grain size is decreased a little. However, for the reason that lower current density causes lower industrial productivity efficiency, the current density for nano-structured black coating electrodeposition is fixed as 3.0 mA/cm^2.

Table 3 Color of nickel film electrodeposited at different current densities (θ=60 °C, pH=3.0, t=30 min, stirring speed= 900 r/min)

Current density/(mA·cm^{-2})	1.0−3.0	4.0	5.0−6.0
Color	Black	Gray	White

The temperature of the electrodeposition solution is varied from 20 °C to 80 °C. As other operating parameters were fixed as pH=3.0, t=30 min, stirring speed=900 r/min, J=3.0 mA/cm^2, the color of the nickel film electrodeposited at different temperatures is shown in Table 4. Since the temperature determines the degree of the molecular thermal motion and viscosity in electrolyte, under the condition of low temperatures (20−30 °C), the dispersibility of electrolyte and the migration rate for Ni^{2+} and supporting electrolyte anion
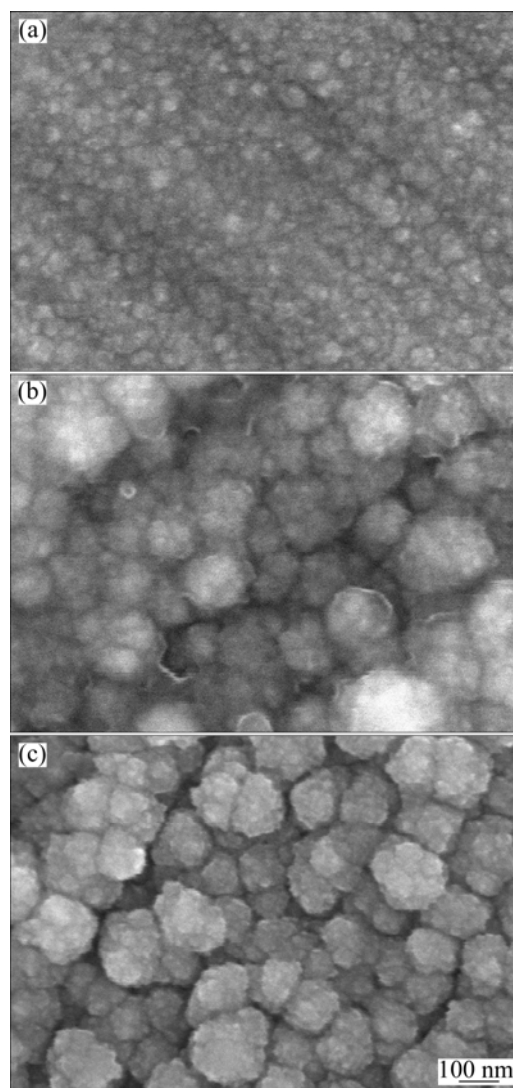
Fig. 2 SEM images of nickel films electrodeposited at different current densities of 1.0 mA/cm^2 (a), 2.0 mA/cm^2 (b), 3.0 mA/cm^2 (c) (θ=60 °C, pH=3.0, t=30 min, stirring speed=900 r/min)

decreased simultaneously, which increased the difficulty for nickel electrodeposition on brass copper substrate[18]. When the temperature rose up to 40 °C and 50 °C, the electrodeposited nickel film showed grayish black in color. At higher temperatures of 60−80 °C, black nickel coatings were electrodeposited. Figure 3 shows the surface morphologies of the black nickel film electrodeposited at 60 °C and 70 °C. It can be clearly seen that the coatings electrodeposited at 60 °C and 70 °C possess nanostructure. However, the film obtained at 70 °C possesses larger grooves than that at 60 °C. Meanwhile, at temperatures higher than 70 °C, the electrodeposition bath becomes unstable due to fast evaporation. Hereinafter, 60 °C was chosen for the nano-structured black nickel film electrodeposition temperature.

Table 4 Color of nickel film electrodeposited at different temperatures (pH=3.0, t=30 min, stirring speed=900 r/min, J=3.0 mA/cm^2)

Temperature/°C	20−30	40−50	60−80
Color	Difficult for Ni electrodeposition	Grayish-black	Black

Table 5 Color of film electrodeposited at different stirring speeds (pH=3.0, t=30 min, θ=60 °C, J=3.0 mA/cm^2)

Stirring speed	600−800	900−1000	1100	>1200
Color	Difficult for Ni electrodeposition	Black	Streaky	Etching
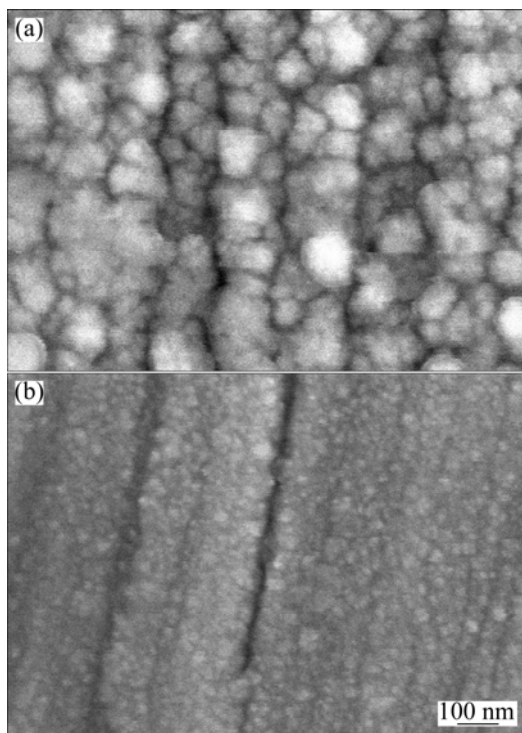
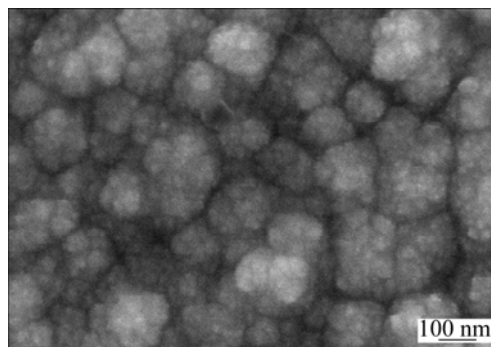**Fig. 3** SEM images of nickel films electrodeposited at 60 °C (a) and 70 °C (b) (pH=3.0, t=30 min, stirring speed=900 r/min, J=3.0 mA/cm^2)

Fig. 4 SEM images of nickel film electrodeposited at stirring speed of 900 r/min (pH=3.0, t=30 min, θ=60 °C, J=3.0 mA/cm^2)

Stirring speed also affects the electrodeposited Ni film color. When the stirring speed was 600−800 r/min, no black nickel coating can be electrodeposited. This indicates that the electrodeposition process is controlled by diffusion effect and the Ni^{2+} prefers to deposit on the surface of formed Ni crystallite [19]. As the stirring speed was raised to 900−1000 r/min, black coatings were electrodeposited. Higher stirring speed such as 1100 r/min will cause streaky coatings, and etching coatings will be electrodeposited at stirring speed above 1200 r/min, as shown in Table 5. Surface morphology of the black nickel coating electrodeposited at 900 r/min is shown in Fig. 4. It clearly shows that the black nickel electrodeposited at 900 r/min possesses nanostructure.

The operating parameters were fixed as pH=3.0, θ=60 °C, stirring speed=900 r/min and J=3.0 mA/cm^2, a lot of experiments were done on the influence of electrodeposition time. When the electrodeposition time was 10−20 min, the film displayed gray color. As the electrodeposition time is prolonged to 30−60 min, black

coatings can be electrodeposited. But electrodeposition time longer than 40 min usually leads to rough appearance. So, the time for uniform black nickel film electrodeposition is fixed as 30 min.

It is known that most black nickel electrodeposition processes is based on an electrolyte containing zinc or cadmium [14,15,20]. In the electrolyte, zinc or (and) cadmium co-deposit with nickel to form black film. However, in this case, the electrodeposition bath does not contain zinc or cadmium. The formation of the black nickel film may originate from the zinc transfer from the brass substrate to the film during the electrodeposition process, the soluble zinc ions generated during the short stewing period of brass copper prior to electrodeposition (which may be not the main reason that the cathode is always galvanized before its immersion in the electrodeposition bath), the nano-structure of the obtained nickel film, or other unclear factors. The formation mechanism of black nickel film in this case is still unknown and further study is in progress to solve this problem.

3.2 Characterizations of optimized black nickel nano-structured film

According to the above study of the technological parameters, the technological parameters of black Ni film electrodeposition were optimized as pH=3.0, θ=60 °C, stirring speed=900 r/min, J=3.0 mA/cm^2. The optimized black Ni film was characterized by SEM and XRD. Figure 5 clearly indicates that the nickel grains are uniform and equi-axed. The magnified image shows that the Ni grain size is in the nanometer range.

Figure 6 shows the XRD pattern of the electrodeposited black Ni film. The diffraction peaks at 44.82°, 52.21° and 76.77° correspond to nickel crystallographic planes of (111), (200) and (220). The average nickel grain diameter can be calculated according to the following Scherrer equation:
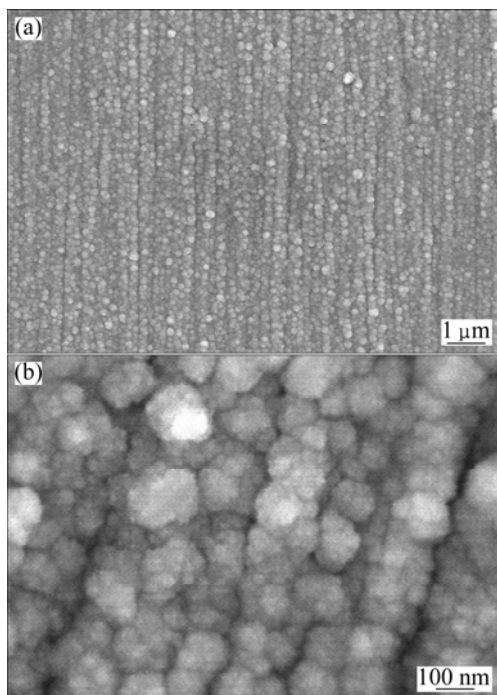
Fig. 5 SEM images of optimized nano-structured black Ni film (pH=3.0, θ=60 °C, stirring speed =900 r/min, J = 3.0 mA/cm^2)
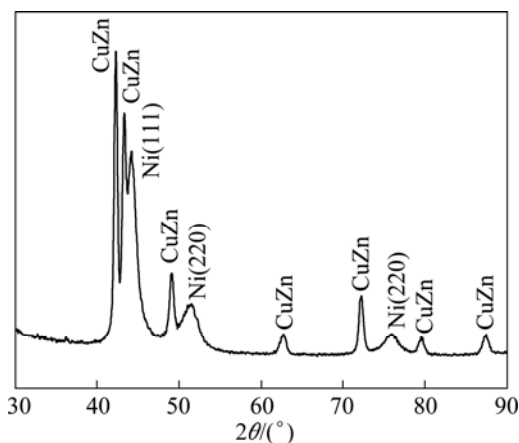
**Fig. 6** XRD pattern of optimized black nano-structured black Ni film

$$L = \frac{K\lambda}{\beta \cos\theta} \tag{1}$$

where K is a constant taken to be 0.89, λ (=0.154059 nm) is the wave length of X-ray used, β is the width at half height of the diffraction peak and θ is the Bragg angle. From the XRD peak intensities, the average diameter of nickel grains is therefore calculated to be approximately

51.4 nm according to the above equation. This calculation result is in agreement with the SEM observation (Fig. 5).

3.3 Corrosion resistance of optimized nano-structured black Ni film

The polarization curve of the nano-structured black nickel film in neutral 3.5% NaCl solutions is shown in Fig. 7. For comparison, the polarization curve of white Ni film is also displayed, which was electrodeposited under the same condition except the variation of the electroplating current density (J=5.0 mA/cm^2). If the current efficiency was calculated on 95%, the thickness of the black nickel and white nickel were 17 μm and 28.3 μm, respectively. Both of them show an active-diffusion transition in the anodic polarization range, but the black nano-structured nickel film shows a lower diffusion current and slightly more negative corrosion potential than the white nickel film, which may be ascribed to the nanostructure of the former. Meanwhile, the corrosion current/potential of black nano-structured nickel film and white nickel film were obtained through the fitting of the polarization curve. For the black nano-structured nickel film, the corrosion current density was calculated as 4.709×10^{-6} A/cm^2, which is much smaller than that of the white nickel film (8.190×10^{-6} A/cm^2). Meanwhile, the corrosion potential of the former (−0.176 V vs SCE) is much higher than the latter one (−0.217 V vs SCE)). This indicates that the black nano-structured Ni film is more resistant to corrosion than the white nickel coating.
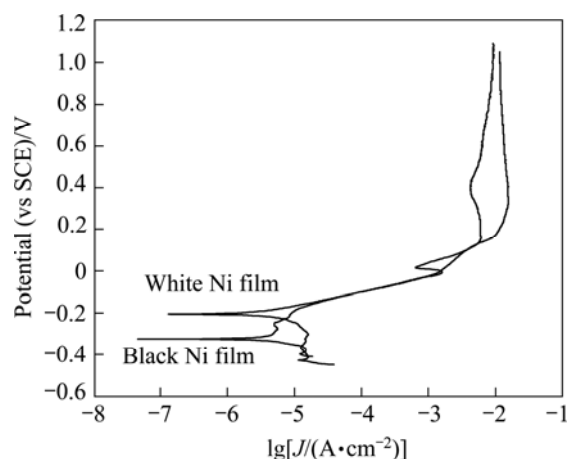

Fig. 7 Polarization curves of nano-structured black Ni and white Ni films in neutral 3.5% NaCl solution

Figure 8 illustrates the EIS plots of the black nano-structured Ni and white Ni films after immersion of 60 min in the neutral 3.5% NaCl solution at open circuit potential (OCP). The time-constant number has been determined through the EIS diagrams (including Nyquist and Bode plots) and the method developed by DEWIT [21,22]. Two EIS plots are similar that they both contain

two capacitive loops in the high frequency range and low frequency range, respectively. The high frequency capacitive loop may be caused by the film, while the low frequency capacitive loop is correlated with the corrosion charge transfer resistance. According to the above analysis, the EIS plots shown in Fig. 8 can be analyzed using two-time constants electrochemical equivalent circuit (EEC) model shown in Fig. 9. In Fig. 9, R_s is the electrolyte resistance; R_f and R_{ct} represent the black (white) Ni film resistance and charge transfer resistance, respectively; and C_f and C_{dl} are associated with the nickel film capacitance and the double layer capacitance. Taking into account the deviation of the capacitance from ideal capacitive behavior, the capacitance in Fig. 9 is replaced by a so-called constant phase angle element, CPE. The impedance of CPE is expressed as

$$Z_{CPE} = 1/[Y_0(j\omega)^n] = \cos(n\pi/2)/(Y_0\omega^n) -$$
$$j\sin(n\pi/2)/(Y_0\omega^n) \qquad (2)$$
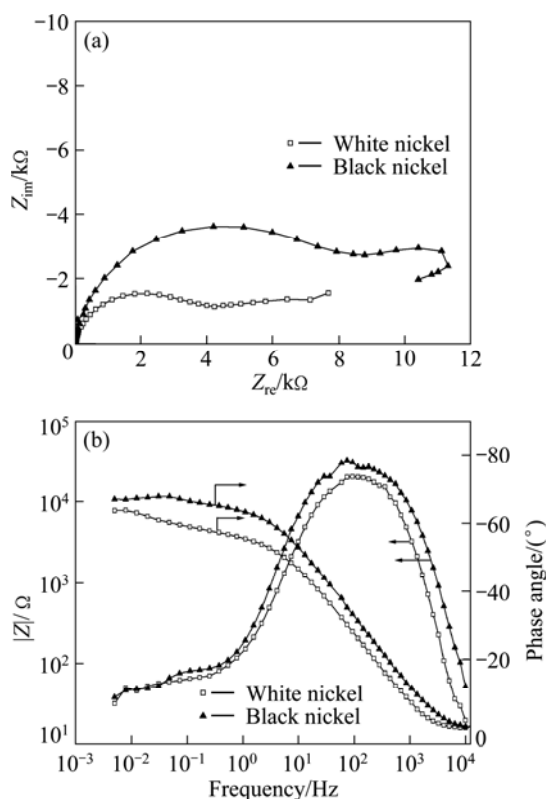
Fig. 8 EIS plots of black nano-structured nickel and white nickel films at open circuit potential immersed in neutral 3.5% NaCl solution for 60 min: (a) Nyquist plot; (b) Bode plot
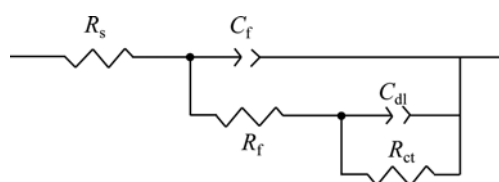
Fig. 9 Equivalent circuit for black and white nickel coating analysis in neutral 3.5% NaCl solution

where ω is the angular frequency of AC-voltage; Y_0 and n are the frequency independent parameters, Y_0 depends on the electrolyte concentration and is connected to the specific electrode capacitance, while the factor n ($0.5 < n < 1.0$) is thought to be characteristic for a given interface and independent of the electrolyte concentration. For $n=1$, CPE represents an ideal capacitance with $C=Y_0$; for $n=0$, a resistance with $Z_{CPE} = Y_0^{-1}$; $n=0.5$, a Warburg; and $n=-1$, an inductance with $L = Y_0^{-1}$. The fitted parameters through the EEC model and Zview program are summarized in Table 6.

Table 6 Fitted EIS parameters of black and white nickel films

Film	$R_s/$ ($\Omega\cdot m^2$)	$CPE_f/$ ($\mu F\cdot cm^{-2}$)	n_1	$R_f/$ ($\Omega\cdot m^2$)	$R_{ct}/$ ($\Omega\cdot m^2$)	$CPE_{dl}/$ ($\mu F\cdot cm^{-2}$)	n_2
Black Ni	15.53	8.1	0.90	6566	2521	6.3	0.93
White Ni	14.24	13.0	0.90	3506	837	48.0	0.45

The charge transfer resistance can reveal the rate of electrochemical corrosion reaction. The smaller the R_{ct} is, the higher the corrosion rate will be. From Table 6, it can be seen that both R_{ct} and R_c of nano-structured black nickel film are much higher than those of white nickel film, and the former is therefore much more associated with corrosion. Meanwhile, lower double layer capacitance (CPE_{dl}) value of the nano-structured black nickel film also indicates its lower corrosion area according to the definition of capacitance (C):

$$C = \frac{\varepsilon S}{d} \qquad (3)$$

where ε is the dielectric constant, S is the superficial area of the film and d is the thickness of the film or the corrosion product. In the case of the nickel film capacitance without corrosion and double layer capacitance of the localized corrosion are indicated respectively as follows:

$$C_{coat} = \frac{\varepsilon_{coat}S_{coat}}{d_{coat}} \qquad (4)$$

$$C_{dl} = \frac{\varepsilon_{dl}S_{dl}}{d} \qquad (5)$$

The more the C_{dl}/C_{coat}, the more serious the localized corrosion is. Based on the above fitted EIS parameter analysis, it can be concluded that the black nano-structured nickel coating possesses a greater corrosion resistance than the white nickel film.

4 Conclusions

1) The color of the electrodeposited nickel film is highly dependent on the technological parameters, including bath pH, temperature, current density, stirring speed and electrodeposition time.

2) The operating parameters were optimized mainly according to the color. The optimized black nickel film possesses nano-structure with an average grain diameter of about 50 nm.

3) The optimized black nano-structured Ni film possesses a higher corrosion resistance than the white Ni film electrdodeposited under the same condition except the variation of the electroplating current density.

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纳米结构黑镍薄膜的电沉积制备及其电化学行为

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摘 要：采用电沉积方法制备具有整体纳米结构的黑镍镀层，并通过肉眼观察结合扫描电镜、X 射线衍射等测试技术研究电沉积过程中的主要参数(电解液 pH、搅拌速度、制备温度及电流密度)对镀层颜色及整体微观结构的影响。进一步采用动电位极化及电化学阻抗等电化学测量技术研究黑镍镀层在中性 3.5% NaCl 溶液中的腐蚀行为及腐蚀机理。结果表明：黑镍镀层的颜色变化趋势决定于电沉积制备参数的选择；通过优化本工艺制备的黑镍镀层平均粒径约为 50 nm。对比了近似条件下制备的光亮镍镀层，发现黑镍镀层在耐蚀性方面具有较大优势。

关键词：电沉积；纳米黑镍薄膜；腐蚀

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