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Electroless Nickel Plating on Fibers for the Highly Porous Electrode

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ABSTRACT :

Materials used as fuel cell electrode should be light, high conductive, high surface area for reaction, catalytic surface and uniformity of porous structure. Nickel is widely used in electrode materials because it itself has catalytic properties. When used as electrode materials, nickel of only a few ìm on the surface may be sufficient to conduct the catalytic role. To manufacture the nickel with porous structure, Electroless nickel plating on carbon fiber be conducted. Because electroless nickel plating is possible to do uniform coating on the surface of substrate with complex shape. Acidic bath and alkaline bathe were used in electroless nickel plating bath, and pH and temperature of bath were controlled. The rate of electroless plating in alkaline bath was faster than that in acidic bath. As increasing pH and temperature, the rate of electrolee plating was increased. The content of phosphorous in nickel deposit was higher in acidic bath than that in alkaline bath. As a result, the uniform nickel deposit on porous carbon fiber was conducted.

Keywords : Electroless plating, Nickel, Carbon, Fiber, Phosphorous

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1. Introduction

Since fuel cells (FCs) have high theoretical thermal efficiencies, FC research and development has been aimed at commercial goals since the discovery of the hydrogen FC concept by Grove in 1839. A FC is an electrochemical cell that converts a source fuel into an electrical current. Molten Carbonate Fuel Cell (MCFC) that is one of the FCs is high temperature fuel cell and it uses nickel for the electrode. [1)] MCFCs have short life time and low efficiency due to non uniform Ni_3Al matrix of electrode. To solve this problem, electroless nickel plating on the carbon fiber that maintains the porous structure was investigated.

Materials used as fuel cell electrode should be light, high conductive, high surface area for reaction, catalytic surface and uniformity of porous structure.[2-4)] Nickel is widely used as electrode materials since nickel itself has catalytic properties. A few μm thickness of nickel on the surface may be sufficient to conduct the catalytic role. The density of carbon fiber is considerably lower than the density of nickel, making it ideal for applications requiring low weight. When carbon fiber is used as substrate for fuel cell electrode the weight can be decreased, drastically. [5)]

Instead of electrical power, reducing agents are used in electroless plating. Electroless plating can make uniform coating on the surface of substrate with complex shape. Electroless nickel plating are widely used in

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chemical, aerospace, automobile and textile industries, due to their ability to provide improved hardness, wear and abrasion resistance.[6-10)]

In this research, electroless nickel plating on carbon fiber was investigated for the uniform deposition in acidic bath and alkaline bath.

2. Experimental Procedures

The substrates used for the electroless nickel plating were the carbon fibers with 8 μm in diameter. For uniform electroless plating and porous structure, the carbon fibers were dispersed in the DI water using sonosmasher. Fig. 1 showed the dispersed carbon fiber by sonosmasher. Since carbon fiber is non-metallic material, sensitization treatment is required prior to the activation treatment. The surface sensitization was conducted by immersion of 10 g/L $SnCl_2$ solution for 5 min. After that, activation treatment was used $PdCl_2$ solution and the samples were cleaned with DI water. The compositions of electroless nickel plating baths were given in Table 1.[6)] The elelctroless nickel plating bath was composed of nickel salt, reducing agent and suitable complex agents. Nickel sulfate was used as nickel source. The hypophosphite was used as reducing agent to both acid and alkaline bath. The complex agents (lactic acid, propionic acid, sodium citrate) were used to buffer the change of pH.

The pH was controlled between 4 and 5 in acidic bath and between 9 and 11 in alkaline bath using NaOH and NH_4OH. Plating temperature was ranged to 40°C-70°C at each pH and the water jacket was used to control the temperature. Energy Dispersive X-ray Spectrometer(EDS) and Field Emission Scanning Electron Microscopy(FESEM) were used for analysis of nickel deposit.

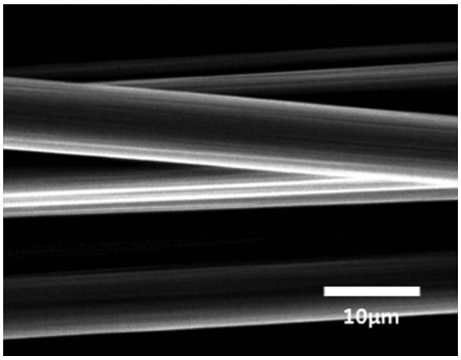


Fig. 1. As received carbon fiber.

Table 1. Compositions of electroless nickel bath (a) acidic bath (b) alkaline bath

(a)

Chemical	Concentration
Nickel sulfate	25 g/L
Sodium hypophosphite	25 g/L
Lactic acid	28 g/L
Propionic acid	2.2 g/L

(b)

Chemical	Concentration
Nickel sulfate	25 g/L
Sodium citrate	50 g/L
Sodium hypophosphite	25 g/L

3. Results and Discussion

In the hypophosphite-based electroless nickel acidic bath, the overall reaction can be written as :[7-8)]

$$Ni^{2+} + H_2PO_2^- + H_2O \rightarrow Ni + H_2PO_3^- + 2H^+ \quad (1)$$

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + H_2 \quad (2)$$

In the hypophosphite-based electroless nickel alkaline bath, the overall reaction can be written as :[7-8)]

$$Ni^{2+} + H_2PO_2^- + 3OH^- \rightarrow Ni + HPO_3^{2-} + 2H_2O \quad (3)$$

$$H_2PO_2^- + OH^- \rightarrow HPO_3^{2-} + H_2 \quad (4)$$

In the above reactions, reactions of (1) and (2) are autocatalytic reaction of nickel. The reactions of (3) and (4) are non-efficient reaction of reducing agent as the reaction proceeds and hydrogen gas are generated.[10)]

From equations at two baths, the reaction rate is influenced by the pH. The nickel deposited carbon fibers in acidic and alkaline bath were shown in Fig. 2 and Fig. 3 respectively. Thickness of nickel was increased with temperature upto 70°C. At the same temperature, surface morphology in acidic bath is smoother than that in alkaline bath. Nickel was not plated at pH 4 and bellow 60°C since energy was not sufficient to overcome activation energy. The deposition rates of acidic bath and alkaline bath were measured and compared in Fig. 4. The rate of electroless plating in acidic bath, pH 5, was 20 nm/min, and that in alkaline bath, pH 11, was 170 nm/min at 60°C. The results indicate that rate of electroless plating in alkaline bath

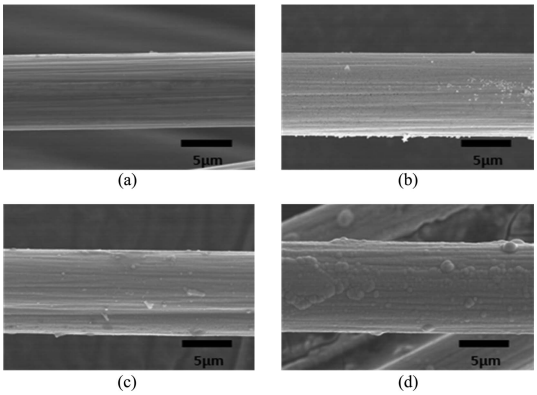


Fig. 2. Electroless nickel deposition on the carbon fiber in acidic bath (a) 40°C (b) 50°C (c) 60°C (d) 70°C.

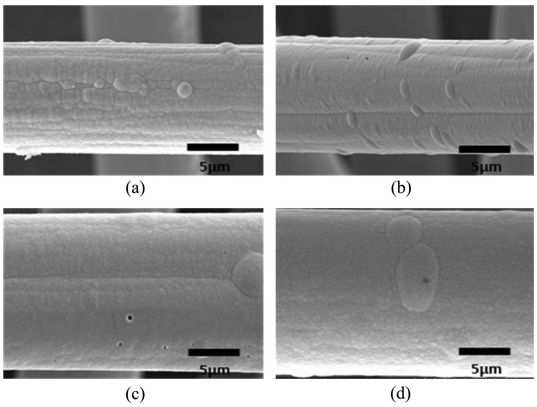


Fig. 3. Electroless nickel deposition on the carbon fiber in alkaline bath (a) 40°C (b) 50°C (c) 60°C (d) 70°C.

was faster than that in acidic bath. As pH was increased in each of the bath, the deposit rate was increased. It can be predicted from the above equations. As pH was increased, the reactions were forced to the forward by Le Chatelier's principle. Secondary reaction also occurs besides above reaction. The reaction is as follow.[7)]

$$H_2PO_2^- + H \rightarrow H_2O + OH^- + P \quad (5)$$

This shows that phosphorus was deposited along with nickel on substrate. The phosphorus contents in the nickel deposit are important to decide the material property. The contents of phosphorous in nickel deposit of acidic bath and alkaline bath were compared in Fig. 5. The content of phosphorous in nickel deposit was higher in acidic bath than that in alkaline bath. As bath temperature was increased, the content of phos-

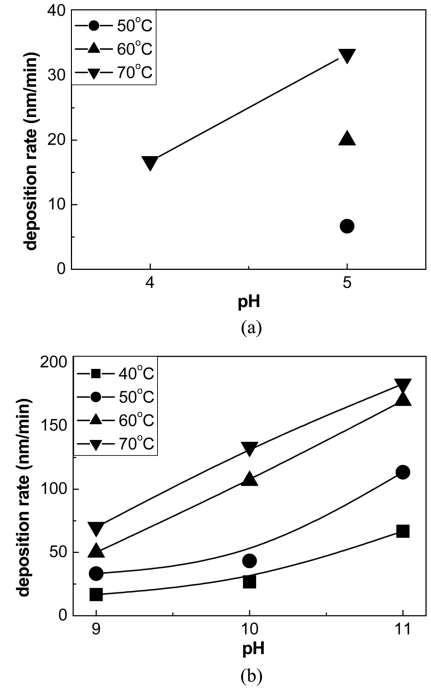


Fig. 4. Effects of pH and temperature on the deposition rate (a) acidic bath (b) alkaline bath.

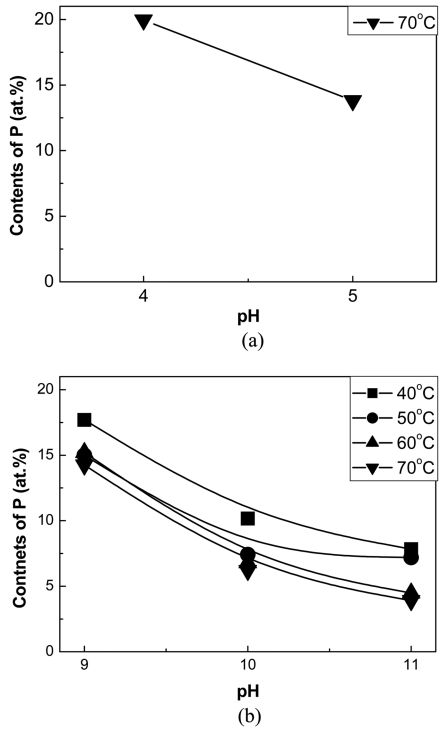
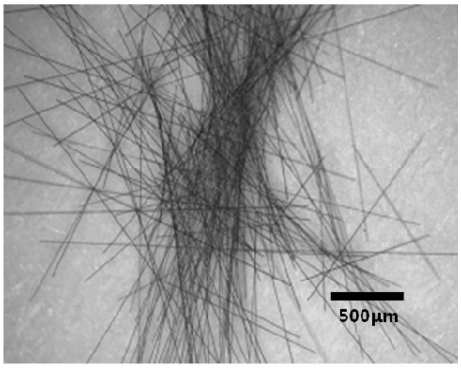
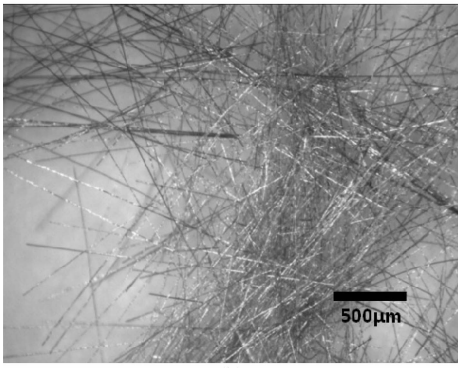


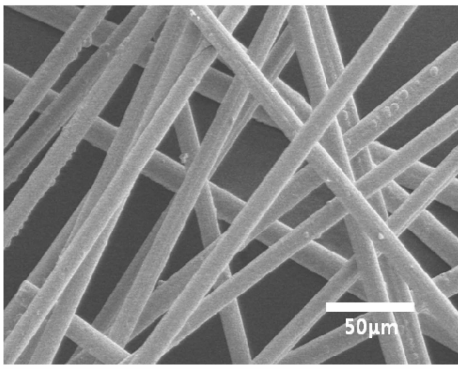
Fig. 5. Effects of pH and temperature on the P content (a) acidic bath (b) alkaline bath.



(a)



(b)



(c)

Fig. 6. Ni on carbon fiber (a) dispersed carbon fiber (b) nickel on dispersed carbon fiber (c) SEM image - nickel on dispersed carbon fiber.

phorous in nickel deposit was decreased. At pH 11, phosphorus contains in nickel deposit is about 4% at 70°C. At pH 5, phosphorous content of deposit was analyzed to above 13% at 70°C.

The nickel covered surfaces of porous structure were shown in Fig. 6. The surface of the carbon fiber substrate was completely covered, and the deposit of nickel was uniform and showed strong adhesion.

4. Conclusions

Electroless nickel plating on the carbon fiber was investigated. The rate of electroless plating in alkaline bath was faster than in acidic bath. The deposit rate of pH 11 is 170 nm/min at 60°C. And concentration of phosphorous in nickel deposit was higher in acidic bath than that in alkaline bath. As a result, using electroless nickel plating the porous structure and light weight was formed with a nickel.

Acknowledgement

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