

## Research Article

# Fabrication of Nickel Nanotube Using Anodic Oxidation and Electrochemical Deposition Technologies and Its Hydrogen Storage Property

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Received 27 January 2016; Revised 29 March 2016; Accepted 24 April 2016

Academic Editor: Biplab K. Debnath

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Electrochemical deposition technique was utilized to fabricate nickel nanotubes with the assistance of AAO templates. The topography and element component of the nickel nanotubes were characterized by TEM and EDS. Furthermore, the nickel nanotube was made into microelectrode and its electrochemical hydrogen storage property was studied using cyclic voltammetry. The results showed that the diameter of nickel nanotubes fabricated was around 20–100 nm, and the length of the nanotube could reach micron grade. The nickel nanotubes had hydrogen storage property, and the hydrogen storage performance was higher than that of nickel powder.

## 1. Introduction

In recent years, great importance has been attached to one-dimensional nanomaterials such as nanowire and nanotube by researchers increasingly due to the unique characteristics and the potential application prospects in the fields of microelectrode [1], single-electron devices [2], chemical sensors, catalysts, and so on [3]. It has been one of the research hotspots to use chemical and physical methods to prepare highly ordered one-dimensional nanomaterials (nanowire and nanotube). For the numerous preparation methods, anodic aluminum oxide template method is widely concerned due to the advantages of low cost, simple technique, uniform aperture structure, high density, and easily controlled aperture parameters. By now, some one-dimensional metal nanomaterials and semiconductive nanomaterials which have polycrystal or single-crystal structures have been fabricated using porous anodic aluminum oxide (AAO) as the template and electrochemical method, such as Au, Ag, Pb, and Si [4–7]. Among numerous one-dimensional nanomaterials, one-dimensional nickel nanomaterials have significant application prospects in the fields of ultrahigh-density magnetic recording media and sensor and nickel-metal

hydride battery due to their excellent magnetic and electrical properties. At present, some researchers have studied the fabrication and performance of nickel nanomaterials [8–11]. However, what is mainly studied is nickel nanowire, and there are few studies on the fabrication and property of nickel nanotube, especially for the study on the hydrogen storage property of nickel nanotube, which is rarely reported. As for the study of hydrogen storage property of nanotube, it is mostly about the study of electrochemical hydrogen storage of carbon nanotube in the current report [12].

Nickel nanotube has the features of high activity, high specific surface area, and hollow structure. It has extensive potential applications. For example, it can be applied to load, dope, and store materials and can also be used as catalyst, hydrogen storage material and sensing element. In this paper, the fabrication of nickel nanotube using porous anodic aluminum oxide as the template was introduced. Besides, the fabricated nanotubes were characterized, and the possible growth mechanisms of nickel nanotube and factors which influence the geometric dimensions were analyzed. Moreover, the nickel nanotubes were made into microelectrode and its electrochemical hydrogen storage property was studied using cyclic voltammetry, the purpose of which was to

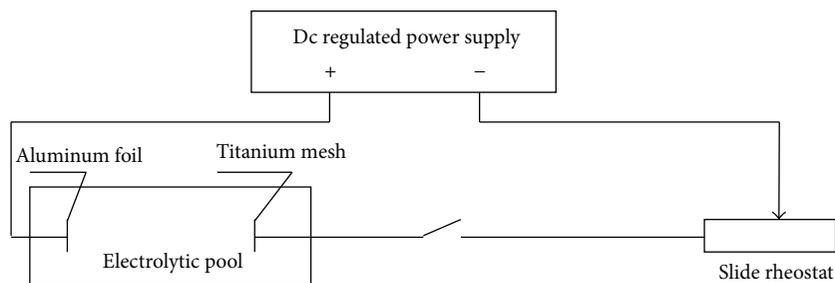


FIGURE 1: The schematic of the preparation of porous anodic aluminum oxide template.

explore and study whether they had application value as nickel-metal hydride battery electrode materials.

## 2. Experimental

**2.1. Preparation of Porous Aluminum Oxide Template.** The 99.99% pure metallic aluminum foil was pretreated by degreasing, removing oxide layer, and rinsing. After pretreatment, metallic aluminum foil was used as anode and oxidized for 30 min under direct voltage. WQI transistor DC stabilized power supply was served as power. The counter electrode was metal titanium mesh, and oxidizing temperature was room temperature. The porous aluminum oxide templates applied in the experiment were all prepared under this condition. The schematic of the experiment is shown in Figure 1.

**2.2. Preparation of Sensitization Solution and Activation Solution.** An appropriate amount of hydrochloric acid was taken to the beaker. Some  $\text{SnCl}_2$  was added into the hydrochloric acid above to dissolve, and then some distilled water was added to dilute the solution so as to prepare  $\text{SnCl}_2$  and HCl sensitization solution with a certain concentration. An appropriate amount of  $\text{AgNO}_3$  was added to some distilled water to dissolve so as to prepare  $\text{AgNO}_3$  activation solution with a certain concentration. The reagents mentioned above were analytically pure.

**2.3. Sensitization and Activation of Porous Aluminum Oxide Template.** The aluminum oxide template was put into the above-mentioned sensitization solution to soak for a few minutes to be sensitized, and then it was washed by deionized water. After washing, the sensitized aluminum oxide template was put into the above-mentioned  $\text{AgNO}_3$  solution to soak for a few minutes to be activated.

**2.4. Electrodeposited Nickel on Aluminum Oxide Template.** A double-electrode system was applied to the electrochemical deposition. The working electrode was aluminum oxide template, and the counter electrode was nickel mesh which was in the same size of working electrode. Electrodeposited solution was a mixed solution that was under a certain proportion of  $\text{NiSO}_4$  and boric acid. WQI transistor DC stabilized power supply (Tianjin Electronic Instruments Plant) was applied in the experiment, and oxidizing temperature was room temperature. The schematic of the experiment is shown in Figure 2.

**2.5. Characterization of Template and Nickel Nanotube Microstructure.** In order to characterize the microstructure and composition of nickel nanotube, chemical approach was applied to separate nickel nanotube from the template. After being nickel-plated, the aluminum oxide template was immersed into 5 wt% NaOH solution to dissolve the aluminum oxide template carrier, and then nickel nanotube powder was obtained. Next, the powder could be washed with distilled water until neutral. XL30 ESEM-TMP environmental scanning electron microscope (Netherlands Philips-FEI Company) (SEM) was applied to observe the surface morphology of aluminum oxide template. Besides, PHILIPS TECNAI G2F20 field emission transmission electron microscopy (TEM) was applied to investigate the morphology and microstructure of nickel nanotube, and the energy disperse spectroscopy was used to analyze the composition of nickel nanotube wall.

**2.6. Fabrication of Nickel Nanotube Powder Microelectrode.** Platinum microwire was sealed in glass capillary by heat sealing, and then the glass capillary was cut off. Besides, the truncation surface was polished and cleaned up with distilled water, ethyl alcohol, and ultrasound successively to form Pt microelectrode. This microelectrode was put into aqua regia which was mildly boiled to corrode the truncation surface of electrode and form a deep microhollow. Then the microelectrode was rinsed with distilled water under ultrasound.

The nickel nanotube powder was tiled on the glass plate, and the microhollow of the truncation surface of microelectrode faced the nickel nanotube powder and was pressed so that the powder could be embedded into the microhollow of microelectrode. After this process, nickel nanotube powder microelectrode was obtained [13].

**2.7. Electrochemical Hydrogen Storage Property.** Cyclic voltammetry test of nickel nanotube microelectrode was conducted on IM6e electrochemical working station which is produced by German Zahner Company. Nickel nanotube powder microelectrode was used as working electrode, NiOH electrode was used as counter electrode, and Hg/HgO electrode was used as reference electrode. Electrolyte was 6 mol/L KOH solution. The scanning range of electric potential was  $-1.60$  V to  $0.00$  V, and the scanning speeds were  $5.0$  mV/s,  $30.0$  mV/s, and  $50.0$  mV/s, respectively.

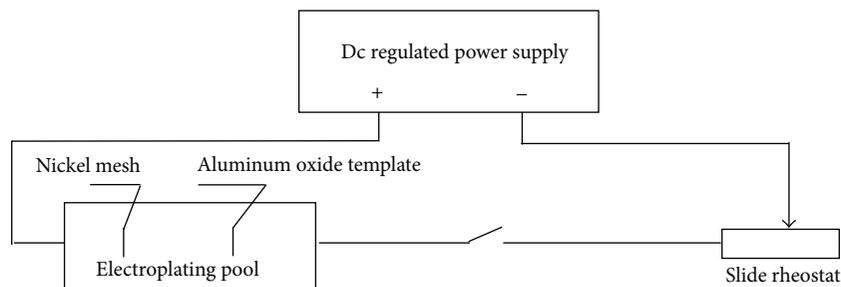


FIGURE 2: The schematic of the preparation of nickel nanotube.

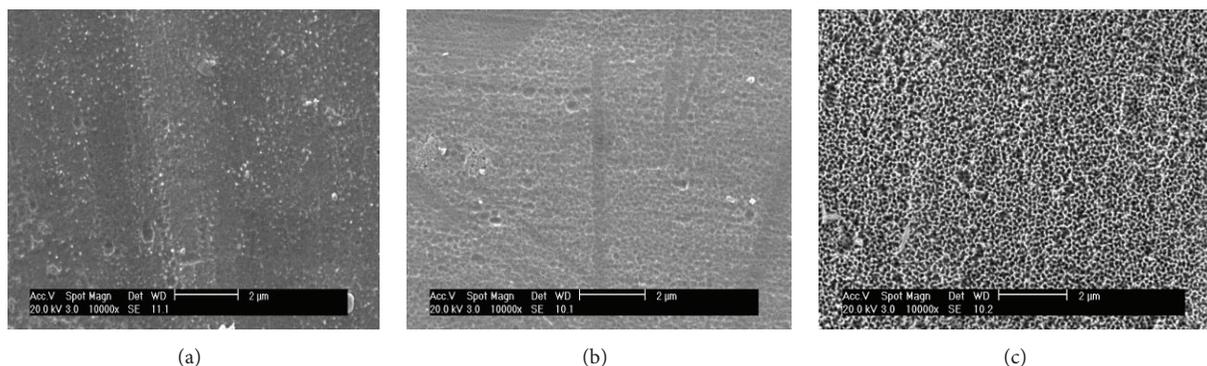


FIGURE 3: The SEM images of anodic alumina oxide templates prepared by different acid solution. (a) Phosphoric acid solution. (b) Sulfuric acid solution. (c) Oxalic acid solution.

### 3. Results and Discussion

**3.1. Influence of Acid Solution.** The SEM images of anodic alumina oxide templates fabricated by different acid solution are shown in Figure 3. It indicates that the acid solution has influence on the porous structure of template in this experimental condition. The surfaces of the AAO template fabricated by phosphoric acid solution and sulfuric acid solution do not show obvious porous structure, which may be because the fact that the acidity of phosphoric acid and sulfuric acid is strong, and when the porous structure is formed, it is dissolved immediately. Thus, deep porous structure cannot be formed on the surface of the AAO template, and it is not appropriate to be used as the template to fabricate nickel nanotube. The acidity of oxalic acid solution is weak, and the dissolution rate of the porous structure in oxalic acid solution is slow, so obvious porous structure can be formed, and it is appropriate to be used as the template for the next step of nickel plating.

**3.2. Influence of Sensitizing and Activating Pretreatment.** The SEM image of AAO template after nickel is deposited on the template without sensitizing and activating pretreatment is shown in Figure 4(a). The porous structure cannot be seen on the surface in Figure 4(a). The SEM image of fracture surface of this template is shown in Figure 4(b). From Figure 4(b), the pores are almost blocked by nickel, and there are a lot of bubbles on the surface. This template was then dissolved by NaOH solution and the powder obtained is analyzed by

transmission electron microscope, as is shown in Figure 4(c). It indicates that only Ni nanowires can be obtained when the AAO template deposited nickel without sensitizing and activating pretreatment, and it is unavailable to obtain tubular structural nickel nanotube. The experimental results show that it is important to have appropriate sensitizing and activating treatment on aluminum oxide template in advance. Otherwise, the deposition surface of template is disordered. A large amount of nickel is accumulated partially that bubbles and cracks resulted. Besides, the porous channels of template are also filled with deposited nickel. Only Ni nanowires can be obtained.

The SEM image of aluminum oxide template before nickel is deposited and after nickel is deposited with sensitizing and activating pretreatment is shown in Figures 5(a) and 5(b). From Figures 5(a) and 5(b), when the aluminum oxide template is processed with sensitizing and activating pretreatment, obvious porous structure can be seen on the surface and the nanoporous channels of template are not blocked by nickel particles. Nickel can grow uniformly on the wall of pores. This is because aluminum oxide template is semiconductor that has poor electrical conductivity, which is not conducive to electrochemical reactions. Thus, before nickel is deposited on aluminum oxide template, reasonable sensitizing and activating pretreatment should be conducted to the aluminum oxide template to guide the growth of nickel in the porous channels of template. The sensitizing process is conducted in acid bivalent stannate solution. The sensitizing process is conducted in acid bivalent stannate solution. When

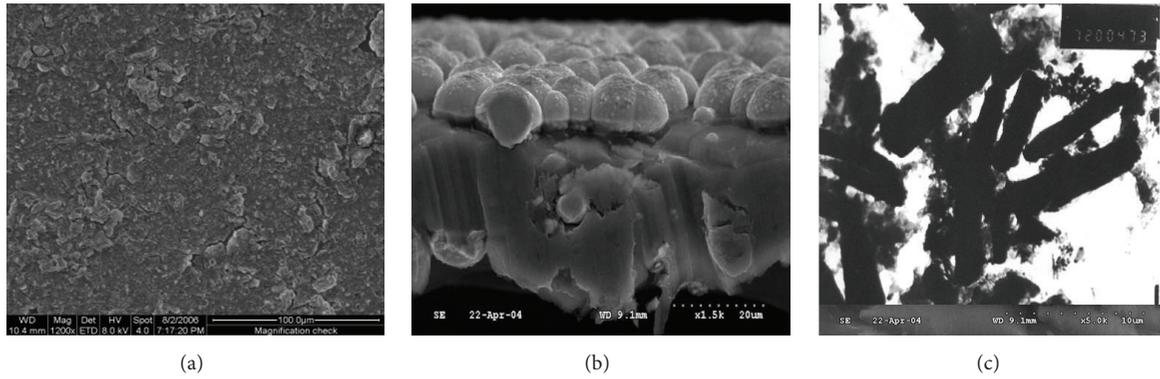


FIGURE 4: (a) The SEM image of AAO template after nickel is deposited without sensitizing and activating pretreatment. (b) The SEM image of fracture surface of AAO template after nickel is deposited without sensitizing and activating pretreatment. (c) The TEM image of the powder obtained by dissolving the AAO template after nickel is deposited without sensitizing and activating pretreatment.

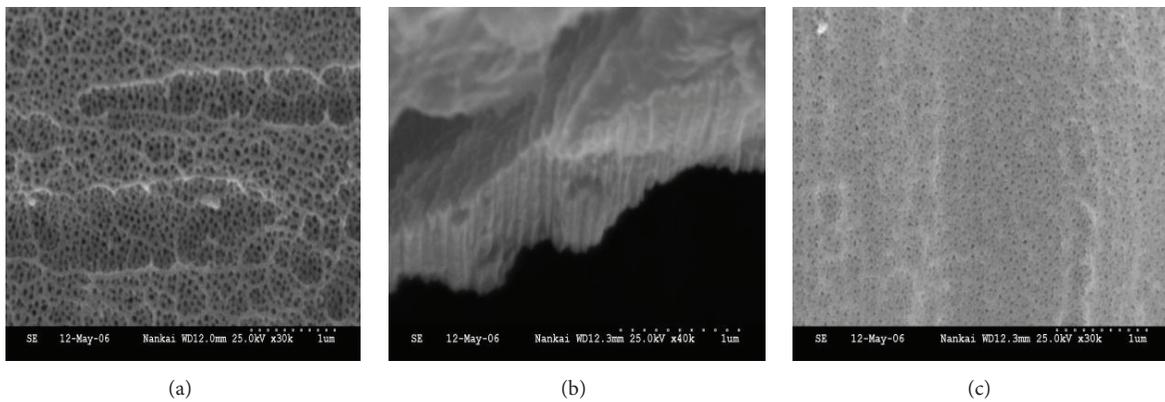
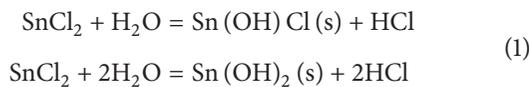


FIGURE 5: (a) The SEM image of AAO template before nickel is deposited. (b) The SEM image of AAO template after nickel is deposited with sensitizing and activating pretreatment. (c) The SEM image of fracture surface of aAAO template after nickel is deposited with sensitizing and activating pretreatment.

the sensitized template is rinsed by distilled water after sensitizing and activating processes, the bivalent tin which is in the porous channels of the template is hydrolyzed. The reaction formulas are as follows:



In water, the bivalent stannate forms tiny gelatinous nanoparticles of basic stannous chloride or stannous hydroxide, and the gelatinous nanoparticles are loose. Thus, in the activated solution, it is easy for  $\text{Ag}^+$  to get into the colloidal particles and be deoxidized to Ag ( $2\text{Ag}^+ + \text{Sn}^{2+} = \text{Sn}^{4+} + 2\text{Ag}$ ) by  $\text{Sn}^{2+}$  immediately. Ag is adsorbed on the colloidal particles to gather into tiny active particles. This adsorption may be a kind of physical adsorption resulted by Van der Waals' force and Coulomb force. These metal nanoparticles will be the deposition core of nickel which guides the growth of nickel.

**3.3. Influence of Current Strength and Length of Deposition Time.** In this experiment, the method of direct current electrodeposition is applied to fabricate nickel nanotube in the nanopores of porous aluminum oxide template. The

experimental results indicate that only when the deposition current and the length of deposition time are controlled properly, the nickel nanotube can be formed. When the deposition current is too high or the length of deposition time is too long, nickel nanotubes cannot be obtained. When the current is too large, only short Ni nanowires can be obtained, as is shown in Figure 6(a). This is because the fact that when the current density is too high, the deposition rate is too fast. Plenty of nickel blocks the nanopores, and it can only form short nanowires. In addition, it is also important to control the length of deposition time. If the length of deposition time is too long, the nickel grows continuously and is filled with the porous channels, and nickel nanotube cannot be fabricated, but only nanowires can be obtained, as is shown in Figure 6(b). According to the experimental results, when the deposition current is in  $8 \text{ mA/cm}^2$  and the length of deposition time is 2 minutes, the nickel nanotubes with stable structure can be fabricated, as is shown in Figure 7.

**3.4. Characterization and EDS Analysis of Nickel Nanotube.** The transmission electron microscope image of nickel nanotube and selected area electron diffraction is shown in

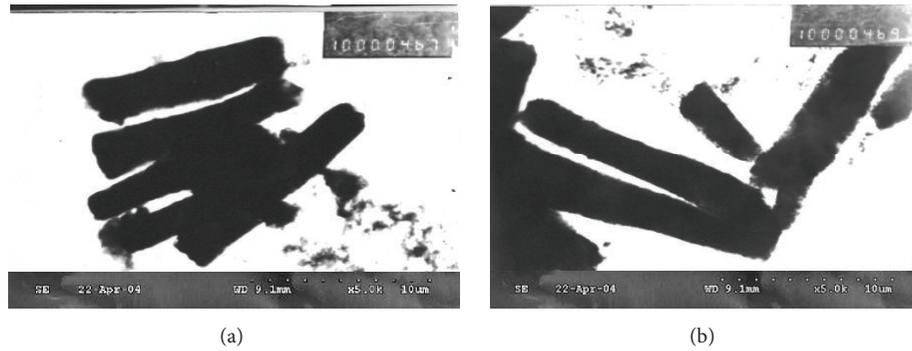


FIGURE 6: Nickel nanomaterial fabricated under different deposition conditions. (a) When the deposition current density is too high ( $20 \text{ mA/cm}^2$ ). (b) When the length of deposit time is too long (10 min).

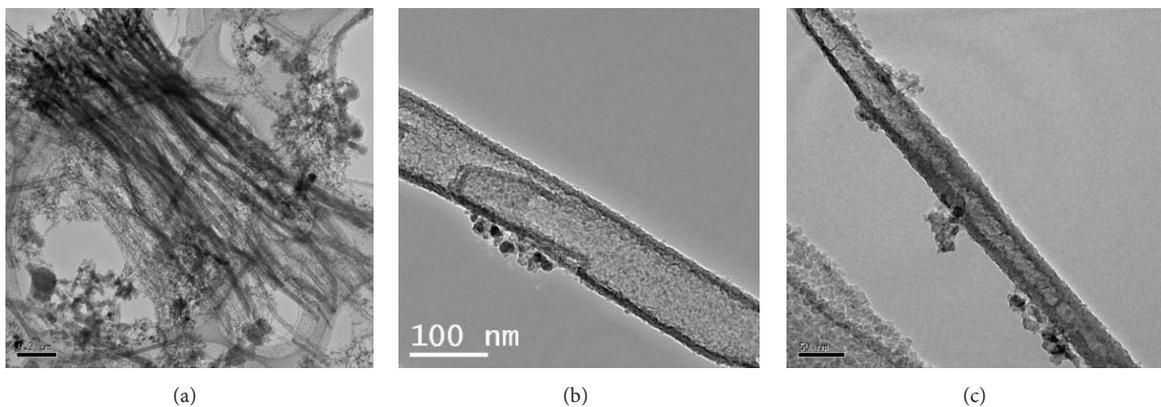


FIGURE 7: The TEM and electron diffraction diagram constituencies of nickel nanotubes. (a) Single nickel nanotube of which the diameter is about 100 nm. (b) Single nickel nanotube of which the diameter is about 20 nm. (c) A cluster of nickel nanotubes.

Figure 7. It indicates that the diameter of nickel nanotube fabricated is nanoscale, around 20–100 nm, and the length can reach micron grade. The thickness of tube wall is uniform. Some nickel nanotubes can be independent, and some nanotubes form clusters. This may be because the fact that the capillary force among contiguous nickel nanotubes, which is the surface tension of water results in, gathers the nanotubes together.

The wall of nickel nanotube is analyzed by energy dispersion X-ray spectrum (EDS) so as to measure the relative content of tube wall elements. The analysis result is shown in Figure 8. As is shown in Figure 8, the main component of tube wall is Ni, and the elements of Al, Ca, S, and C are brought in by AAO template. The existence of Cu is due to the fact that the screen mesh of transmission electron microscope is copper mesh. Second, there is a small quantity of Ag in the tube wall which is brought in by  $\text{AgNO}_3$  from the activating solution. It means that Ag of activating solution gets into the porous channels of porous aluminum oxide template and is adsorbed on the internal surface of tube wall, which creates conditions for the subsequent electrodeposition reactions.

**3.5. Cyclic Voltammetry Analysis of Nickel Nanotube Micro-electrode.** Hydrogen storage materials which can absorb and release hydrogen reversibly are the main negative electrode

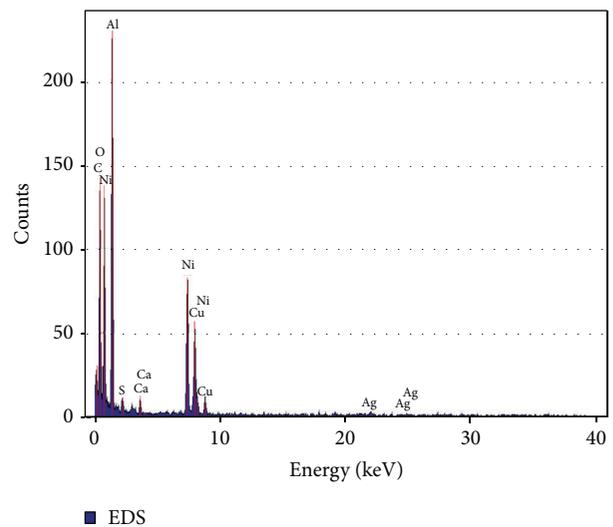
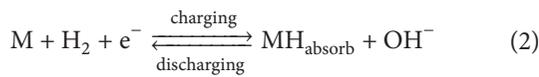


FIGURE 8: The EDS figure of nickel nanotubes.

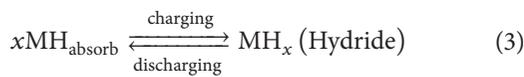
materials of nickel-metal hydride battery. If the interior of nickel nanotubes can also absorb and release hydrogen reversibly, it is hopeful to apply nickel nanotube to the field of nickel-metal hydride battery.

The nickel nanotube powder and commercially available nickel powder are made into microelectrode and used as working electrode to conduct cyclic voltammetry test, respectively. The battery absorbs and releases hydrogen in the process of charging and discharging with charge migration. Hydrogen storage electrode reaction is a multistep reaction, which includes charge transfer reaction and several material transfer (diffusion of  $\text{H}_2\text{O}$  and  $\text{OH}^-$  and diffusion of H atoms inside the nanotube powder), which can be expressed as follows:

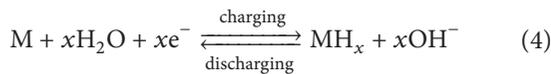
Charge transfer reaction is



Diffusion of H atoms inside the nanotube powder is



Overall electrode reaction is



The hydrogen storage capacity of nickel nanotube and nickel powder can be mirrored from the volume of charge migration. The capacity of the hydrogen storage electrode is directly proportional to the amount of hydrogen stored in the hydrogen storage material; the greater the hydrogen storage capacity is, the greater the electrode capacity is. Correspondingly, in the cyclic voltammetry, the larger the peak area is, the higher the peak current is.

The cyclic voltammetry curves of nickel nanotube and nickel powder microelectrode and the cyclic voltammetry curve of hollow Pt microelectrode were tested at different scanning speeds, respectively, which is shown in Figure 9. As is shown in Figure 9, there is significant difference between the cyclic voltammetry curve of powder microelectrode and hollow microelectrode, where it is demonstrated that nickel nanotube and nickel powder are already loaded into the microelectrode. For the same microelectrode, the cyclic voltammetry curves of electrodes are similar at different scanning speeds, which indicated that the values of oxidation peak position and adsorption peak position of hydrogen are similar for the same electrode at different scanning speeds. The difference is the value of peak current which is greater correspondingly when the scanning speed is higher. Thus, the curve at a certain scanning speed can be used as an example to be analyzed. Take the scanning speed at 50 mV/s, for example, as is shown in Figure 9, with regard to nickel powder microelectrode, the oxidation peak position is among the voltage range of  $-0.56\text{ V}$  to  $-0.66\text{ V}$  (reference electrode is Hg/HgO, and the following is the same), and the adsorption peak position is among the voltage range of  $-0.90\text{ V}$  to  $-1.00\text{ V}$ . Compared with nickel powder microelectrode, the

adsorption peak position moves around  $0.36\text{ V}$  negatively, which is demonstrated that the adsorption of hydrogen on nickel nanotube microelectrode is delayed compared with the adsorption of hydrogen on nickel powder microelectrode. The reasons may be as follows: nickel powder and nickel nanotube have difference on microstructure. Nickel powder consists of small particles. The hydrogen atoms can only be adsorbed on the surface of particles. However, nickel nanotube has hollow cavity. Hydrogen atoms are diffused in the cavity and adsorbed on the internal wall. Thus, as for nickel nanotubes, when the potential negatively moves to the voltage range of  $-0.90\text{ V}$  to  $-1.00\text{ V}$  which is also the adsorption peak position of nickel powder, hydrogen atoms are only diffused into the cavity of tubes without adsorption. At this time, adsorption peak cannot be seen. When the potential negatively moves to the voltage range of  $-1.26\text{ V}$  to  $-1.36\text{ V}$ , the hydrogen atoms diffused into the cavity of nickel nanotube reach a certain quantity. At this time, hydrogen atoms are adsorbed on the surface of nickel nanotubes' wall to form an adsorption peak. Therefore, the adsorption peak position of nickel nanotube microelectrode moves negatively compared with nickel powder electrode. However, for the nickel nanotube microelectrode, the oxidation peak position of hydrogen moves positively because the hydrogen atoms stored in the cavity need be diffused to the surface first, and then they can be oxidized to form an oxidation peak. However, nickel powder only adsorbs hydrogen atoms on the surface of particles. It can be oxidized without diffusion. Thus, the oxidation peak position of nickel nanotube has positive move compared with the oxidation peak position of nickel powder. These indicate that nickel nanotubes have hydrogen storage property. Besides, compared with nickel powder, the hydrogen storage property of nickel nanotube is better.

The cyclic voltammetry curves of nickel nanotubes at different scanning speed were shown in Figure 10. The patterns of cyclic voltammetry curves at different scanning speeds are similar, which indicates that the influence of scanning speed on the mechanism of the electrode reaction is negligible. However, the peak potential difference of the nickel nanotube electrode increases with the increase of scanning speed. As for completely reversible electrode reaction, the peak potential difference has nothing to do with the scanning speed, so the nickel nanotubes electrode reaction is not completely reversible. However, with the scanning speed range from  $5\text{ mv/s}$  to  $50\text{ mv/s}$ , the increase of peak potential difference is not significant, which indicates that the nickel nanotubes microelectrode reaction has good reversibility to some extent.

#### 4. Conclusions

- (1) The nickel nanotube which has a stable structure can be fabricated by the method of electrochemical deposition using porous aluminum oxide as the template. The diameter of nickel nanotube fabricated is around  $20\text{--}100\text{ nm}$ . The thickness of the tube wall is uniform, and the length of the nanotube can reach micron grade.

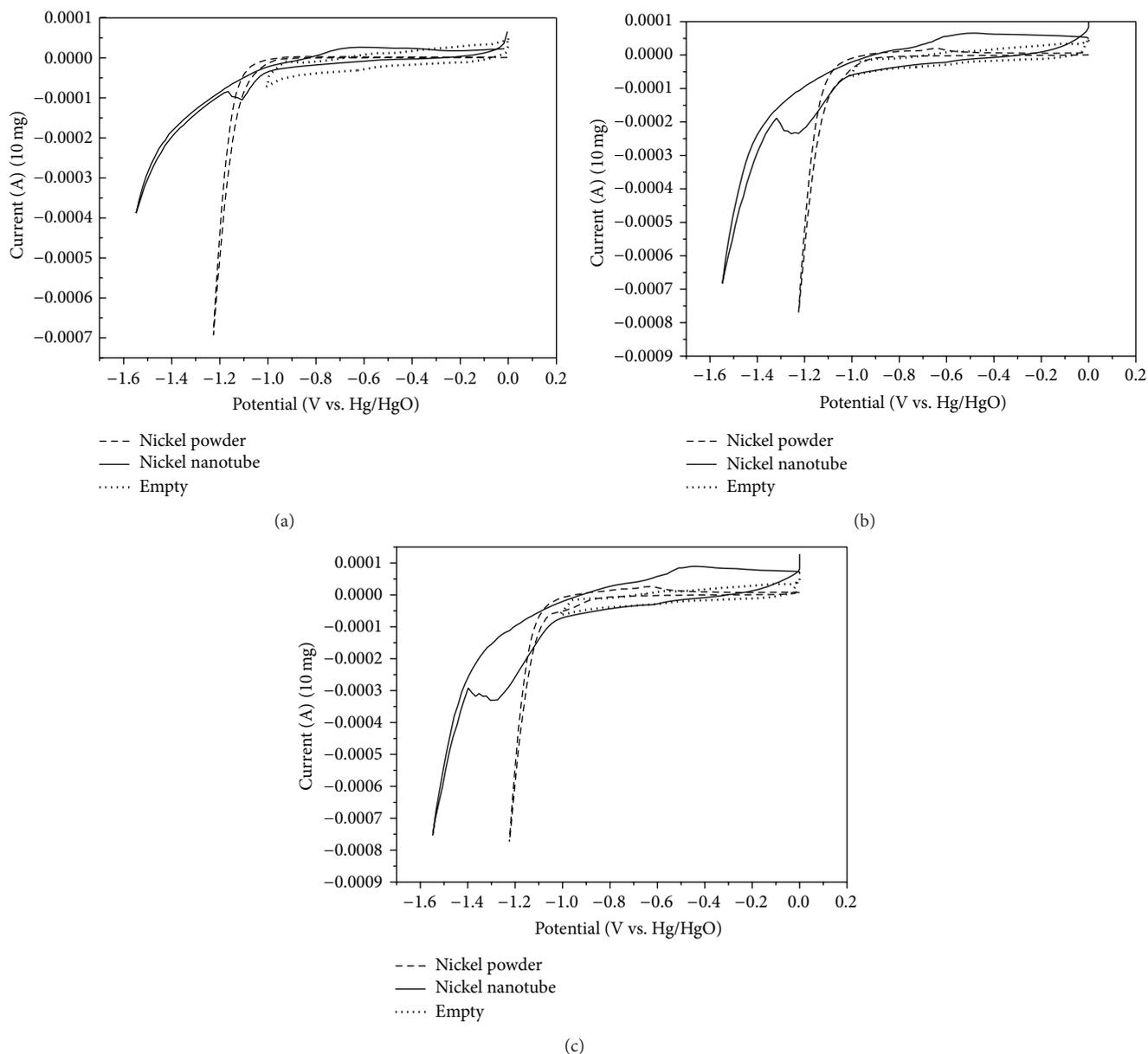


FIGURE 9: The cyclic voltammetry curves of nickel nanotubes and nickel powder microelectrode and empty Pt microelectrode at different scanning speed. (a) Scanning speed 5 MV/s. (b) Scanning speed 30 MV/s. (c) Scanning speed 50 MV/s.

- (2) Acid solution has an obvious influence on the surface structure of porous aluminum oxide template. The acidity of oxalic acid solution is suitable, which is an ideal electrolyte to fabricate porous aluminum oxide template.
- (3) The sensitizing and activating pretreatment of aluminum oxide template and the current strength and length of deposition time are the key factors which influence the growth of nickel nanotube.
- (4) The possible growth mechanism of nickel nanotubes can be speculated as follows: through the activating

and sensitizing process, Ag ions get into the porous channels of porous aluminum oxide template and are adsorbed on the internal surface of tube wall, which can provide reaction center for nickel deposition, and nickel can grow continuously along the tube wall until nanotubes are formed.

- (5) The effect of electrochemical hydrogen storage of nickel nanotubes is superior to that of nickel powder. Compared with nickel powder, the electrochemical oxidation reaction of nickel nanotube is easier to happen, and the reactivity is also higher.

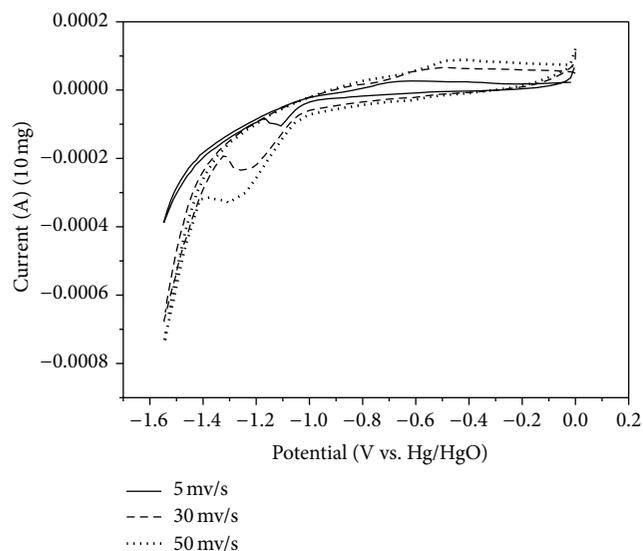


FIGURE 10: The cyclic voltammetry curves of nickel nanotubes at different scanning speed.

## Competing Interests

The author declares that there is no conflict of interests regarding the publication of this paper.

## Acknowledgments

The author appreciates experimental assistance of Zheng Chen.

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