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Influence of boric acid (H_3BO_3) concentration on the physical properties of electrochemical deposited nickel (Ni) nanowires

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Abstract. Authors have investigated the influence of the stabilizer (Boric Acid) concentration during the template-assisted electrochemical deposition of Nickel (Ni) nanowires in Anodic Alumina Oxide (AAO) templates. The synthesis was performed using Ni Sulfate Hexahydrate ($NiSO_4 \cdot 6H_2O$) as metal salts and Boric Acid (H_3BO_3) as a stabilizer. The mixture of both solutions creates electrolyte and utilized for the electrochemical deposition of Ni nanowires. During the experiment, the boric acid concentration varied between 5 g/L, 37.5 g/L and 60 g/L with a deposition temperature of 80 °C (constant). After the electrochemical deposition process, AAO templates were cleaned with distilled water before dissolution in Sodium Hydroxide (NaOH) solution to obtain the freestanding Ni nanowires. Physical properties of the synthesized Ni nanowires were analyzed using Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive Spectroscopy (EDX) and X-ray Diffraction (XRD). The physical properties of obtained Ni nanowires has elaborated by taking into account the effect of boric acid concentration on the surface morphology, growth length, elemental composition and crystal orientation crystal of the synthesized nickel nanowires. The finding exposes that the boric acid concentration does not influence all aspects in the physicals properties of the synthesized Ni nanowires. The boric acid concentration did not affect the surface texture and crystal orientation. However, shorter Ni nanowires obtained as the concentration of boric acid increased.

1. Introduction

Synthesis of nanostructures with defined morphology and properties has attracted serious attention among nanotechnology researchers due to the importance of the dimensionality which classifies their properties [1, 2]. Among various available method, template-assisted electrochemical deposition is one of the popular and well-utilized approaches [3]. This method is relatively straightforward [4] with controlled diameter, cost efficient and allows the duplication of complex topology present on the surface a template in a single step [5, 6]. The method typically operates at ambient temperatures and pressure as well as can be used for mass production of nanostructures with controlled geometry and morphology [2, 7-9]. Additionally, among templates, Anodic Alumina Oxide (AAO) templates are the most widely used due to its effective medium for synthesis of metallic nanowires. These templates have an advantage over controlling the diameter of nanowires precisely and producing relatively straight nanowires [7, 10-15].



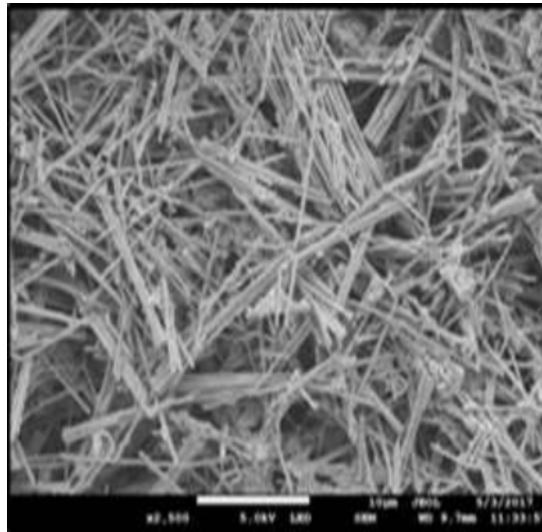


Figure 1. Synthesized Ni nanowires.

Ni nanowires with their ferromagnetic properties are of interest in numerous applications due to their lower cost, and they can be consistently synthesized via electrochemical deposition [16]. The properties of synthesized nanowires are significantly dependent on the synthesis parameters, such as solution pH, deposition bath temperature, magnetic field [17] and current density [18]. During the experiment, the boric acid concentration was varied between 5g/L, 37.5g/L, and 60 g/L to study the effect of different boric acid concentration during the synthesis on their physical properties. Boric acid is one of the important chemical compounds in the electrolyte for Ni deposition since they act as buffering agents in an electrolyte [19]. The nanowires then characterized by using Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive Spectroscopy (EDX) and X-ray Diffraction (XRD). The influence of boric acid concentration on surface texture, growth length, elementary composition and crystal orientation on synthesized Ni nanowires discussed in greater detail.

2. Experimental details

Alumina oxide membranes (Whatman, 99.9% pure) was used in this template-assisted electrochemical deposition. The synthesis procedure was carried out using a two-electrode electrochemical cell system. One side of AAO templates coated with an aluminum coating which acts as a cathode and Ni plate which serves as an anode, placed into electrodeposition bath, parallel to each other. When an electric field is applied, cations diffuse through the channels and deposit on the cathode, resulting in the growth of nanowires inside the pores of the template. For the electrolyte solution preparation, the procedure adopted by Ertan et al. [20] was used. It is by far the simplest method and has demonstrated success in synthesizing Ni nanowires using the template-assisted electrochemical deposition method. 71 g of Ni metal salt, (Ni Sulfate Hexahydrate, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$) dissolved in 400 mL distilled water. In a separate beaker, boric acid (H_3BO_3) dissolved in room temperature in distilled water. The two solutions were mixed and stirred for 30 minutes at 25 °C until the solutions well mixed. The correlation between the stabilizer agents concentration and the physical properties of Ni nanowires, the amount of boric acid used for synthesis had been manipulated at 5 g/L, 37.5 g/L, and 60 g/L respectively while the amount of Ni sulfate hexahydrate kept constant. The electrochemical bath cell was placed on top of a magnetic stirrer and heater from Heidolph Instruments GmbH & Co. The whole system was then placed inside the fume hood to limit the exposure of hazardous gas. The Keithley systems source meter was used to supply the required electrical current for electrodeposition. The applied current was set at 3 mA, and during the electrochemical deposition process, the current monitored for continuous and constant deposition. The sudden fluctuation (increase or decrease) in current gives an indication of the inconsistent growth of nanowires, and the process needs to be ended

to prevent the disproportionate growing of the nanowires and also to avoid the collapse of the pore walls due to extreme growth pressure. The electrochemical deposition time duration is set to be 60 minutes (constant for all sample). As a deterrent, during the electrodeposition process, it is vital to ensure the copper tape used as the electrical connector on both cathode and anode are not soaked into the electrolyte to avoid contamination to the nanowires. Table 1 shows the simplified synthesis condition used for the experiment. After the electrodeposition of Ni nanowires, 10 g of sodium hydroxide (NaOH) dissolved in 15 ml distilled water. Then, etching process takes place by placing the electrodeposited AAO template inside NaOH solution. The whole mixture rapidly shook until the AAO template completely dissolved and a dark grey product (Ni nanowires) formed at the bottom part of the glass bottle. The dark grey products then cleaned using ethanol solution several times by magnetic decantation and ready for characterization.

Table 1. Synthesis condition of electrochemically deposited Ni nanowires.

Experiment	Boric Acid Concentration (g/L)	Electrochemical Deposition Time (min)	Applied Current (mA)
A	5	60	3
B	37.5	60	3
C	60	60	3

3. Results and discussion

Ni nanowires have been successfully deposited on the AAO templates. The influence of boric acid concentration on physical properties of obtained Ni nanowires investigated. In the conventional Ni plating, the electrolyte solution is known as Watts bath, which composed of Ni sulfate and Ni chloride as metal salts and boric acid act as buffering agents. Holm and O'Keefe demonstrated Ni plating using electrolyte solution without boric acid and produced brittle deposition of Ni due to the precipitation of Ni hydroxide on the substrate surface [21]. The surface appearance improvement can be performed by increasing the activity of Ni either by increasing the metal salt concentration or temperature or decreasing the solution pH. In the next section, the influence of boric acid concentration on surface morphology of Ni nanowires is discussed.

3.1. Influence of boric acid concentration on surface morphology

FESEM (JOEL JSM-7800F) used to investigate the morphology of the synthesized Ni nanowires. The analysis performed by qualitative observation on the surface synthesized Ni nanowires from a sequence of images taken using FESEM. The surface morphology imaging conducted at a beam source of 5kV, and a working distance of 10 mm. Fig. 2 (a-b) show the FESEM images of surface morphology Ni nanowires electrodeposited using boric acid concentration of 5g/L. Fig. 2 (c-d) and Fig. 2 (e-f) shows the FESEM images of surface morphology of Ni nanowires using boric acid concentration of 37.5 g/L and 60 g/L respectively.

From the FESEM images, it is observed that there are no significant differences in surface texture of the synthesized Ni nanowires. All the surface texture found to be rough and flaky. These needs further examination to precisely conclude the finding. However, from the study, the appearance of the Ni deposit was greatly improved with the presence and increased the concentration of boric acid. Instead of a brittle, irregular surface, a smooth, glassy nickel surface formed with the presence of the boric acid. The improved surface structure reported being attributed to the buffering ability of boric acid [22, 23]. In this present study, the observation obtained found in contrast with the previous finding reported and appears that the influence of boric acid on the surface texture of electrodeposited Ni nanowires remains complicated unclear.

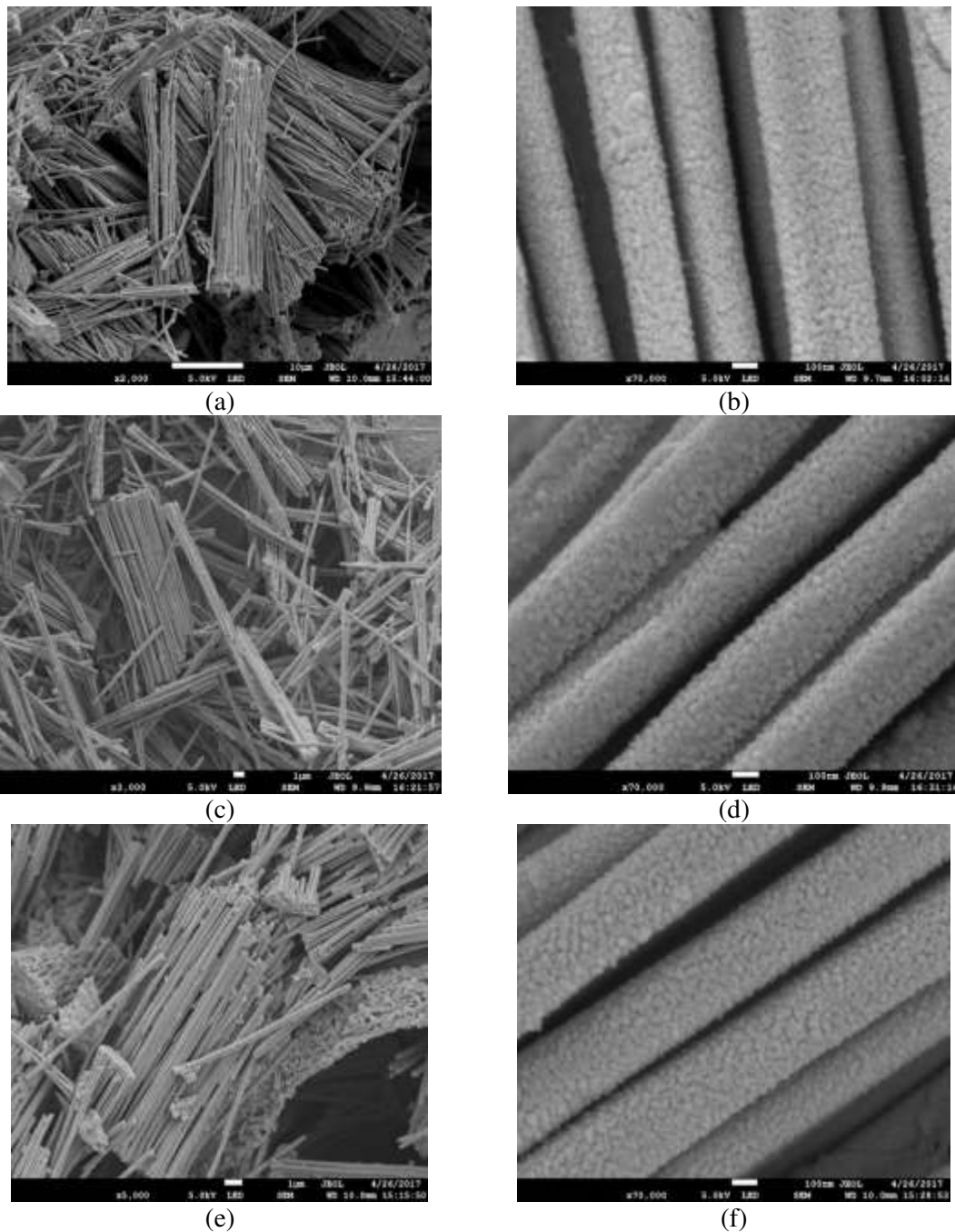


Figure 2. FESEM images of Ni nanowires synthesized at different boric acid concentration: (a-b) 5 g/L boric acid concentration, (c-d) 37.5 g/L boric acid concentration, (e-f) 60 g/L boric acid concentration.

3.2. Influence of boric acid concentration on growth length

The growth length of Ni nanowires at different boric acid concentration was measured using FESEM. Average ten (10) length measurements taken for each synthesis condition. The measured growth length of electrodeposited Ni nanowires at all boric acid concentration used for synthesis tabulate in Table 2. The finding used to investigate the influence of boric acid concentration on the growth length of the Ni nanowires.

The growth length of Ni nanowires found to decrease significantly as the concentration of boric acid increased. These are due to the presence of a high concentration boric acid which inhibits the Ni ion deposition [23].

Table 2. Measured length of grown Ni nanowire at different synthesis condition.

Boric Acid concentration	Ni nanowires Length (μm)
5g/L	33.125
37.5g/L	15.850
60g/L	12.600

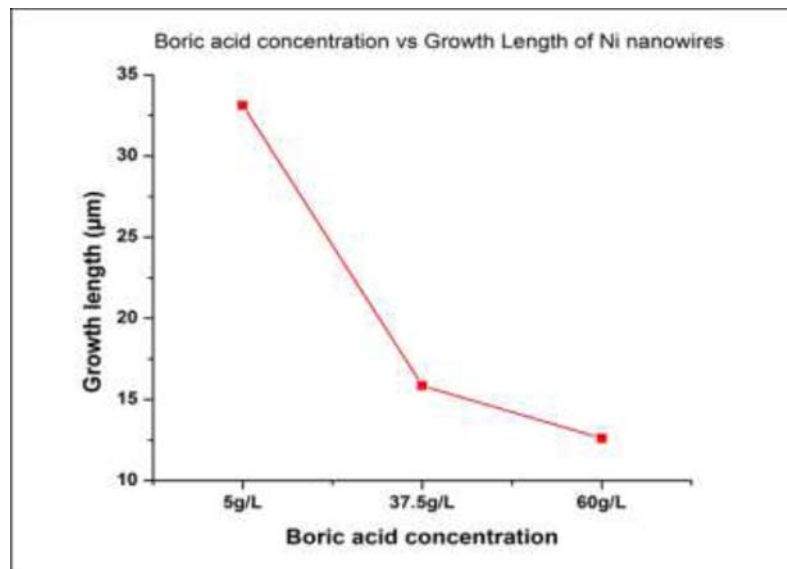


Figure 3. Nickel Nanowire grown length at different boric acid concentration.

3.3. Energy Dispersive X-Ray (EDX) analysis

A quantitative EDX analysis is performed on all the samples to determine the elemental composition of the grown Ni nanowires deposited on the AAO templates at different boric acid concentration and the results are shown in Table 3. The EDX analysis performed using Oxford Instruments EDX which attached to JOEL JSM-7800F FESEM. The experiments carried out at a beam source of 15kV. The EDX analysis demonstrates that the synthesized Ni nanowires were consistently composed of 97.83 % of Ni and 2.17 % of Oxygen. The presence of small amount of oxygen in the entire spectrum indicates potential absorption from the air on the surface of Ni nanowires.

Table 3. EDX elemental composition of synthesized Ni nanowires.

Elements	Value (%)	Standard deviation (%)
Nickel (Ni)	97.83	0.4
Oxygen (O ₂)	2.17	0.4

3.4. Influence of boric acid concentration on crystal orientation

Figure 3.2 shows the XRD spectrum of Ni nanowires synthesized at different concentration of boric acid. The XRD spectrum shows there was no significant influence of boric acid concentration on the orientation of the crystals. The predominant orientation is still dictated by the concentration of the

metal salts. The preferred orientation of synthesized Ni nanowires in all cases was found to be (1 1 1) plane, followed by (2 0 0) and (2 2 0) planes.

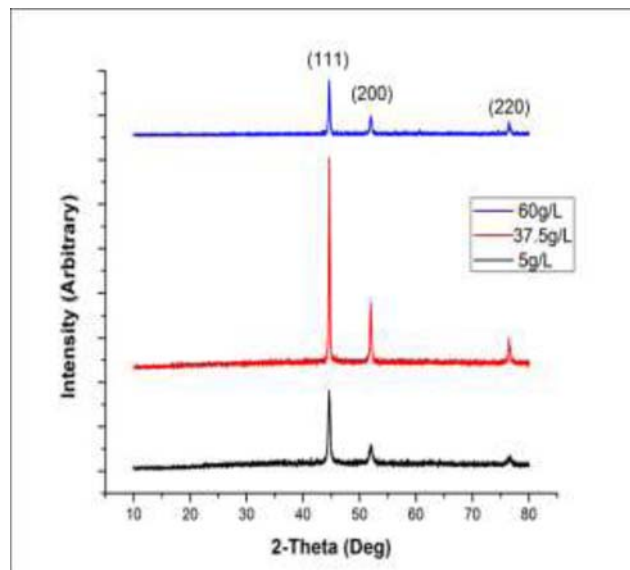


Figure 4. XRD spectrum of Ni nanowires synthesized at a different boric acid concentration.

However, the spectrum of Ni nanowires synthesized at a boric acid concentration of 37.5 g/L presents the highest intensity of diffraction peaks demonstrating a better crystalline structure. From the obtained trend of XRD spectrum, the optimum concentration of boric acid to synthesize the best crystallite structure could be suggested at 37.5 g/L. The obtained spectrum further used to derive the 2-theta peak angle which is used to calculate the spacing between atoms for the nanowires synthesized at a boric acid concentration of 5 g/L, 37.5g/L, and 60 g/L. Table 4 presents the calculated d-spacing for Ni nanowires synthesized at this process parameters as compared with bulk Ni literature data. Bragg's formulation was used to determine d-spacing for the deposited Ni nanowires. From the table, we can clearly see that there are no significant differences between the bulk nickels d-spacing from the literature with the present study.

Table 4. 2θ angle and d-spacing for nickel nanowires synthesized at different deposition temperature compared with bulk nickel literature data.

Sample	(1 1 1)		(2 0 0)		(2 2 0)	
	2θ (deg)	d (Å)	2θ (deg)	d (Å)	2θ (deg)	d (Å)
Ni bulk (literature)	44.37	2.04159	51.596	1.77	76.0884	1.25098
Ni nanowires						
5g/L	44.620	2.02836	51.979	1.75716	76.533	1.24329
37.5g/L	44.639	2.02754	52.003	1.75641	76.487	1.24393
60g/L	44.627	2.02805	51.976	1.75725	76.683	1.24124

4. Conclusions

As a conclusion, Ni nanowires has been successfully synthesized using the template-assisted electrodeposited technique. The properties analysis reveals that boric acid concentration does not influence all the aspects. From the FESEM analysis, for all the investigated cases, the surface texture found to be rough and flaky. The observation obtained was in contrast to the previous finding and appears that the influence of boric acid on Ni deposition is complicated and remains unclear. Where else, in the growth length, from the ten (10) length measurements taken for each boric acid condition,

the growth length of Ni nanowires is found to decrease significantly as the concentration of boric acid increased. These are due to the presence of a high concentration boric acid which inhibits the Ni ion deposition. The EDX analysis demonstrates that the synthesized Ni nanowires were consistently composed of 97.87 % of Ni and 2.17 % of Oxygen. The small amount of oxygen present in the entire spectrum indicates the possible absorption from the air on the surface of Ni nanowires. The XRD spectrum also shows there was not a significant influence of boric acid concentration on the preferred orientation of the crystals. The predominant orientation still primarily dictated by the concentration of metal salts. The preferred orientation of synthesized Ni nanowires in all cases was found to be (1 1 1) plane, followed by (2 0 0) and (2 2 0) planes

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