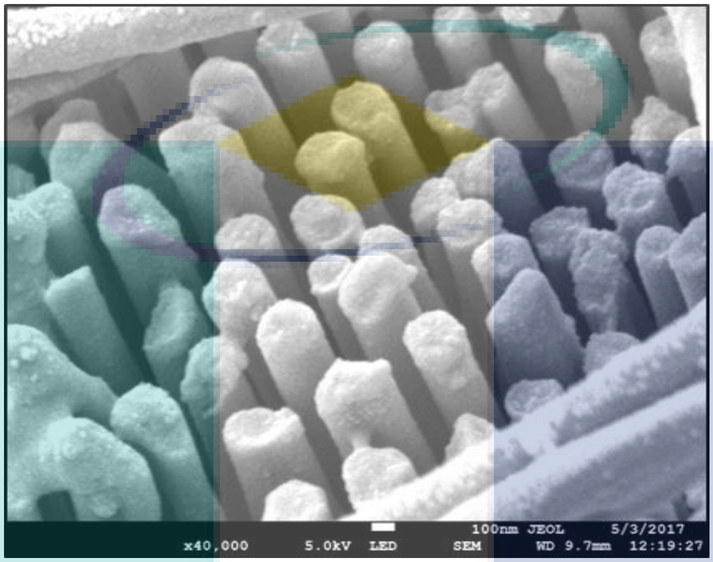
LAMPIRAN B

TEMPLATE
BUKU PROFIL PENYELIDIKAN SKIM GERAN PENYELIDIKAN GERAN UNIVERSITI JANGKA PENDEK / GERAN DALAM UMP



EFFECT OF SOLUTION CONCENTRATION AND TEMPERATURE ON THE GROWTH OF FERROMAGNETIC NANOWIRES DURING ELECTRODEPOSITION

Name of Project Leader
ASSOC. PROF. IR. TS. DR. MAHENDRAN SAMYKANO

Name of co-researchers
PROF. DR. MD. MUSTAFIZUR RAHMAN
DR. NURUL AKMAL BINTI CHE LAH
DR. NGUI WAI KENG
DR. RAMLI JUNID

DEPARTMENT OF MECHANICAL ENGINEERING, COLLEGE OF ENGINEERING
Mahendran@ump.edu.my

RDU170320

ABSTRACT (120 words)

The present research and dissertation focuses on: 1. Experimental synthesis of electrodeposited Ni nanowires at different solution concentration and temperature, 2. Physical properties characterization of the synthesized nanowires to understand their morphology, structural and crystallographic properties. Key research insights from the present experimental research include: Electrodeposition method consistently synthesizes high purity Ni nanowires with a significant improvement in surface morphology when optimum value of solution concentration applied, during synthesis; X-ray diffraction (XRD) characterization and analysis indicate that solution concentration and heating temperature has significant influence on the crystal orientation of Ni nanowire.

1. INTRODUCTION

The distinctive ability of nanomaterials, i.e., the Ni nanowires, as future technological applications in science, technology, and engineering forced the scientist and researchers to study, investigate and further enhance the current state of knowledge on these nanowires. Novel experimental procedures and techniques are in continuous search to characterize these metallic nanowires and also the mechanism to extract the results accurately. Before any application of the material can be realized, it is crucial to have the ability control the synthesis and perform the appropriate characterization technique to understand the behavior and capability of the structure in interest. Thus, it is important to study the behavior of these structures starting from the synthesis at all the possible configuration and with well-defined characterization technique to understand the behavior of the material in interest to the fullest extent. These would help in the determination of the capability and the possible prospect of application. In the present research, 1D Ni nanowires are synthesized using AAO template-assisted electrodeposition method. The synthesis performed at different electrodeposition bath temperature and boric acid concentration (stabilizer). The synthesized Ni nanowires then subjected to various physical characterization to understand the influence of the electrodeposition parameter towards the physical properties.

2. RESEARCH METHODOLOGY

Table 1 Deposition process parameters used for synthesis

Electrolyte	Solution Concentration	Heating Temperature(°C)
A	5 g/L	40 80 120
`B	37.5 g/L	40 80 120
C	60 g/L	40 80 120

As the objective of the study to synthesize and investigate the influence of electrodeposition bath temperature and boric acid concentration on the physical properties of electrodeposited Ni nanowires, the amount of boric acid and bath temperature are varied while other parameters kept constant (Refer Table 1). All synthesis was done for 1 hour of deposition at a current density of about 3.0 mA. After the electrodeposition process, the templates were cleaned with deionized water prior to dissolution in sodium hydroxide (NaOH) solution to obtain freestanding Co nanowires. NaOH solution was later replaced with methanol solution.

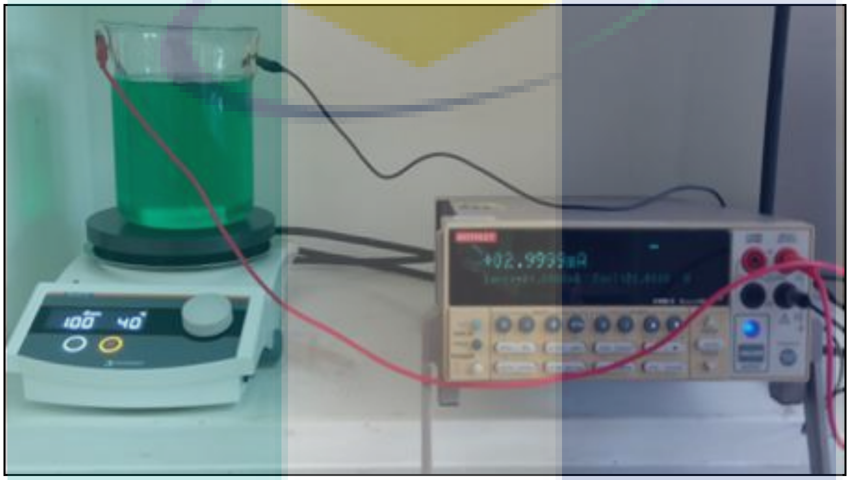


Figure 1 Electrodeposition set-up for Ni nanowire synthesis

3. LITERATURE REVIEW

It is a well-established fact that the electrodeposition synthesis process in porous templates initiates at the bottom edge of the cathode (negative) surface of the pore. The high surface area and the presence of sites with low coordination number in the porous part of the alumina afford energetically favorable sites for initiating metal adsorption during electrodeposition [1]. The metal ion M^{n+} is transported from the electrolyte into the ionic metal lattice. Meanwhile, electrons are provided from the external electron source (power supply) to the electron gas of the metal M. This electrodeposition process illustrated in Figure 1a. Miguel García et al. [2] describes that during the electrodeposition process, the template pores are initially filled with a liquid precursor from the chemical reaction of the electrodeposition process. The solidification of the filled liquid precursor takes place to form nanowires as shown in Figure 1b. These are a very simple route, but it is critical to ensure that the pores are filled with the fluid. If the solution has an excellent wettability towards the template, it can diffuse through the membrane producing an enrichment of the solid component in the interior of the pores. Once the pores filled, the template is removed from the solution and dried. The mechanisms of the pore filling are different for various approach and template.

4. FINDINGS

From the FESEM analysis, we can claimed that well-defined and excellent repeatability of Ni nanowires have been generated inside the pores of AAO templates on the AAO templates with uniform diameter (200 nm – 330 nm).

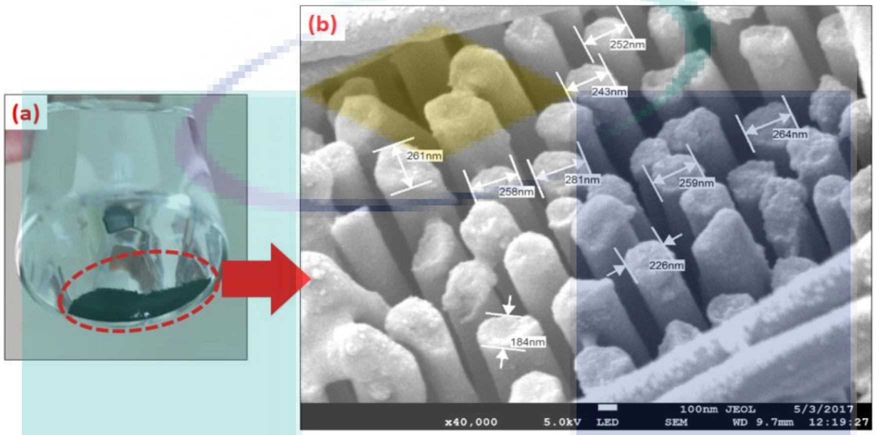


Figure 1 (a) Obtained Ni nanowires; (b) Diameter measurement of synthesized Ni nanowires by FESEM analysis.

Elementary Composition -The EDX characterization proves that the Ni nanowires were composed of 96.99 % of Ni and 3.01 % of O_2 (Figure 3a and Figure 3b). The small percentage of O_2 observed indicates the possible absorption of O_2 from the air on the surface of Ni nanowires.

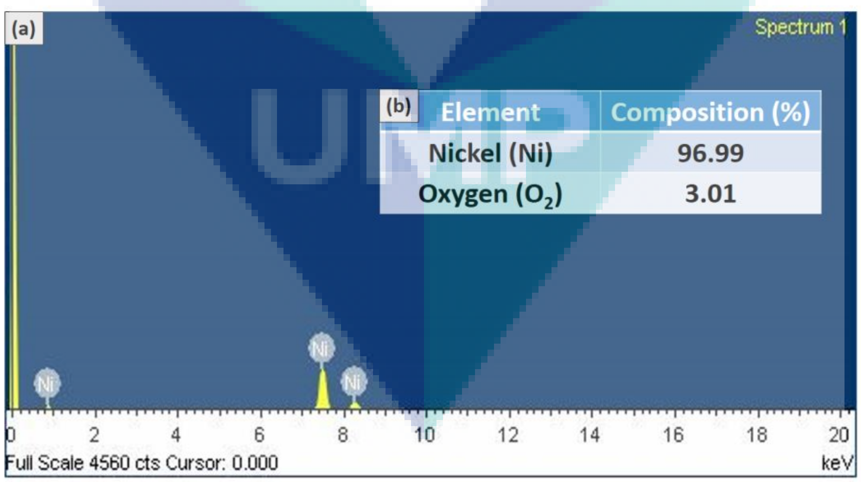


Figure 2 EDX spectrum of synthesized Ni nanowires

Surface texture - From the FESEM analysis, it is noticed that all the synthesized Ni nanowires have rough and flaky surface texture. Also, the observation found that there are significant differences when the parameter of the synthesis condition changed. All the surface texture become rougher with the increasing of stabilizer agent concentration. This is due to the increasing of internal stress which cause the cracks on the nanowires surface texture. This possible reason can be supported by Davolas et al., research findings; presence of boric acid in electrochemical deposition process cause the surface texture of deposited Ni to be smooth and glassy, instead of brittle and irregular surface appearance [3]. Boric acid serve as buffering agents for electrochemical deposition[4], and important for hasten the formation of nanowires. However, the present research findings found to be opposite with the literatures and the correlation of stabilizer agent concentration to the surface texture of Ni nanowires remains complex and ill-defined.

Additionally, the surface texture change to be very rough when the heating temperature increased to 110 °C. This is due to the formation of greater grain size at the high deposition temperature. The consequence from this phenomena, large Ni grains are obstructed to generate inside the templates pore during the formation of nanowires[5].

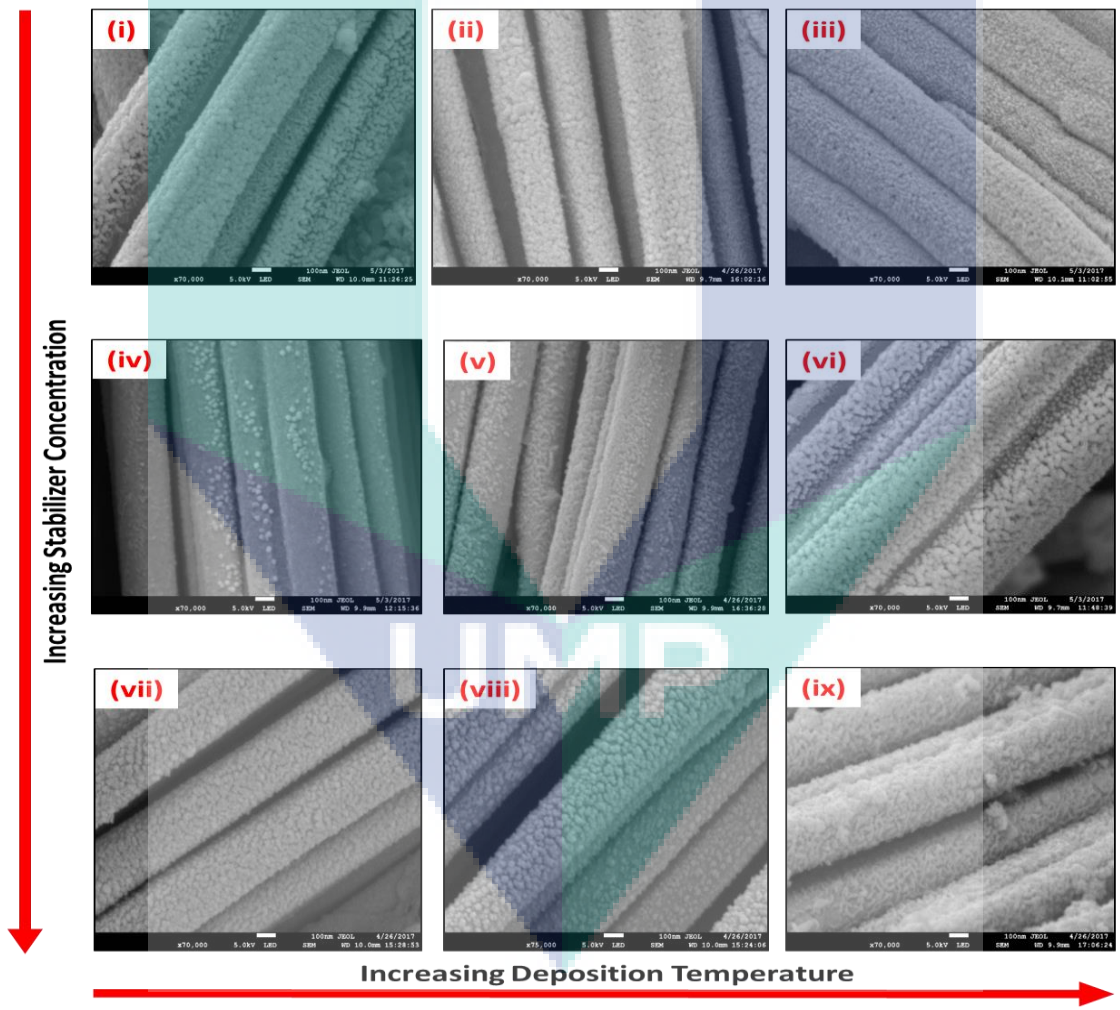


Figure 3 FESEM analysis of synthesized Ni nanowires.

Growth length -Figure 5 shows the average measured growth length of the synthesized Ni nanowires for all parameters. The length of Ni nanowires found to be longer when heating temperature set to be high. Higher temperature increase the rate of atomic diffusion[6], increase the speed of Ni nanowires formation and thus accelerates growth relative to the nucleation of new grains [7]. The influence of deposition temperature to the growth length of Ni nanowires also due to the lowered energy barriers [8]. However, the longer nanowire growth will creates a higher center of gravity which makes their position unbalanced [9].

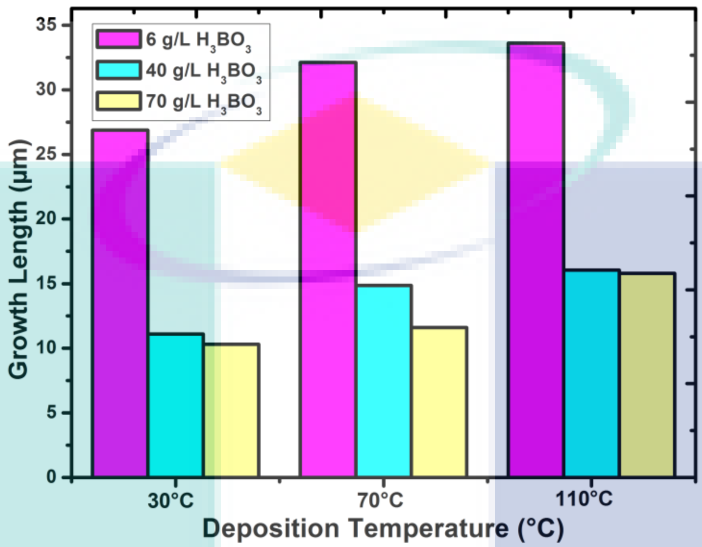


Figure 4 Ni nanowire length for different synthesis condition

Crystal Orientation- XRD Analysis

Figure 6 show the XRD spectrum for the synthesized Ni nanowires at all synthesis condition. The diffraction patterns for all synthesis condition exhibited the synthesis Ni nanowires are polycrystalline as the crystalline planes with Miller indices of 111, 200, and 220. The relative intensity of Ni (111,200 and 220) peak increase as the deposition temperature increase showing that the quality of crystalline was increased with the higher deposition temperature [10].

In the aspect of boric acid concentration, it is observed that 40g/L of boric acid concentration with deposition temperature of 110 °C obtained the highest diffraction peak followed by 6g/L and 70g/L cases. These imply that 40g/L boric acid concentration give the best crystal quality to the synthesized Ni nanowires. It can be concluded that stabilizer agent concentration influence the crystal quality of Ni nanowires with the ideal stabilizer agent concentration of 40 g/L with a heating temperature of 110 °C.

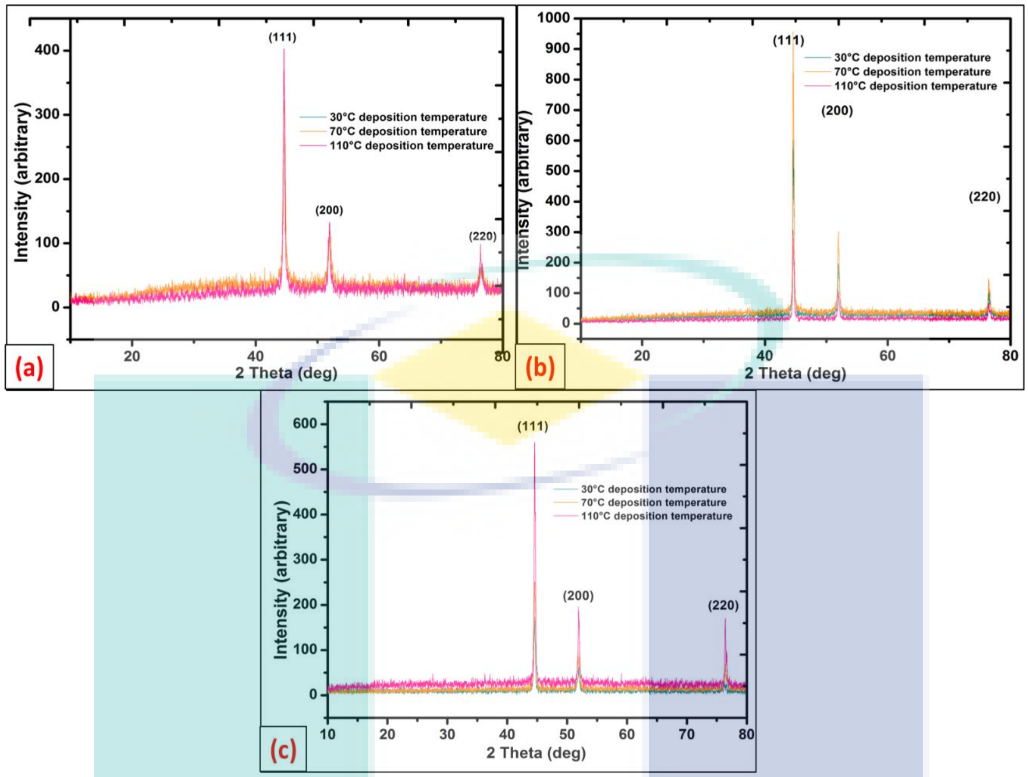


Figure 5 XRD characterization

Crystal size - The results strongly suggest that the crystal size improves as the heating temperature set to be higher. This indicates that higher temperature would exhibit the crystalline structure of the Ni nanowires due to the favouring growth of pre-existing nuclei during the initial stages of the electrochemical mechanism [6].

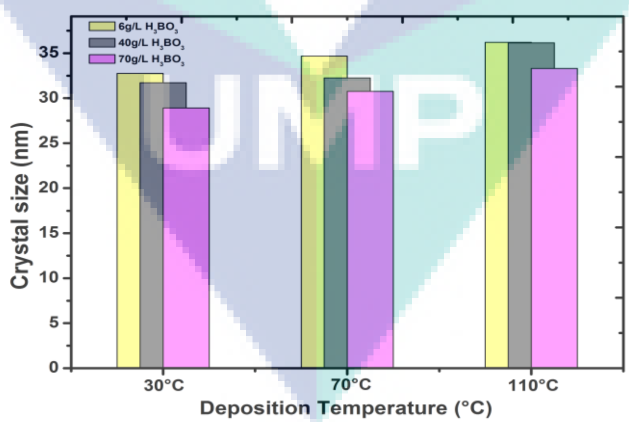


Figure 6 Crystal size of Ni nanowires synthesized at different stabilizer agent concentration and heating temperature

Discussing the influence of stabilizer agent concentration to the crystal size of nanowires, the crystal size of synthesized Ni nanowires found to be slightly decrease as the stabilizer agent become more concentrated. Further analysis needed to figure out the possible causes behind these hypothesis. Vicenzo and Cavallotti revealed that when boric acid added to the electrolyte at pH above 4, they extends the stability field of the crystallographic and improves its quality of crystals produced [11]. Another literature claimed that the reducing of boric acid concentration cause the increase in pH value, thus decrease the current efficiency (100% to 63%)[12].

5. CONCLUSION

Well-defined and excellent repeatability of Ni nanowires have been successfully deposited on the AAO templates with uniform diameter in the range of 200 nm – 330 nm via template-assisted electrochemical deposition approach. The obtained Ni nanowires with high purity (97.97% of nickel based on the EDX result) have rough and flaky surface texture for all the synthesis condition cases. Observation from FESEM analysis also claim that the surface texture roughness increased as the heating temperature increased. There are significant differences in the surface texture observed when stabilizer agent concentration increased. Overall growth length found to be greatly improved as the heating temperature increase but decrease when stabilizer agent concentration is high. The diffraction patterns for all synthesis condition exhibited the synthesis Ni nanowires are polycrystalline as the crystalline planes with Miller indices of 111, 200, and 220. The relative intensity of Ni (111,200 and 220) peak increase as the deposition temperature increase showing that the quality of crystalline was increased with the higher deposition temperature. It is also noted that the average of crystal size increase when stabilizer agent concentration decrease. Where else, the crystal size increased when the heating temperature increased. Further investigation needed to understand the rational reason for the characterization of obtained Ni nanowires influence by stabilizer agent concentration and heating temperature.

ACHIEVEMENT

i) Name of articles/ manuscripts/ books published

- Mechanical properties–Theory and experiment. Applied Materials Today, 11,320-337.
- Plasmonic behaviour of phenylenediamine functionalised silver nanoparticles. Materials Research Express, 4(9), 095018
- An overview of marine macroalgae as bioresource. Renewable and Sustainable Energy Reviews, 91, 165-179.

ii) Title of Paper presentations (international/ local)

- Nanofluid as coolant for grinding process: An overview. In IOP Conference Series: Materials Science and Engineering (Vol. 342, No. 1, p. 012078). IOP Publishing.
- Electrochemical deposited nickel nanowires: influence of deposition bath temperature on the morphology and physical properties. In IOP Conference Series: Materials Science and Engineering (Vol. 257, No. 1, p. 012032). IOP Publishing.
- Influence of boric acid (H3BO3) concentration on the physical properties of electrochemical deposited nickel (Ni) nanowires. In IOP Conference Series: Materials Science and Engineering (Vol. 257, No. 1, p. 012033). IOP Publishing

iii) Human Capital Development

- Santhi A/P Ulakanathan (MSM17004) – Expected Graduation in 2020

REFERENCES

1. Narayanan, T.N., et al., *On the growth mechanism of nickel and cobalt nanowires and comparison of their magnetic properties.* Nano Research, 2008. **1**(6): p. 465-473.
2. García, M., P. Batalla, and A. Escarpa, *Metallic and polymeric nanowires for electrochemical sensing and biosensing.* TrAC Trends in Analytical Chemistry, 2014. **57**: p. 6-22.
3. Davalos, C., et al., *Study of the role of boric acid during the electrochemical deposition of Ni in a sulfamate bath.* Int J Electrochem Sci, 2013. **8**: p. 9785.
4. Ji, J., et al., *Surface pH measurements during nickel electrodeposition.* Journal of Applied electrochemistry, 1995. **25**(7): p. 642-650.
5. Toimil-Molares, M.E., *Characterization and properties of micro- and nanowires of controlled size, composition, and geometry fabricated by electrodeposition and ion-track technology.* Beilstein Journal of Nanotechnology, 2012. **3**: p. 860-883.
6. Cortés, A., et al., *Single-crystal growth of nickel nanowires: influence of deposition conditions on structural and magnetic properties.* Journal of nanoscience and nanotechnology, 2009. **9**(3): p. 1992-2000.
7. Wang, J.-G., et al., *Controllable template synthesis of superconducting Zn nanowires with different microstructures by electrochemical deposition.* Nano letters, 2005. **5**(7): p. 1247-1253.
8. Dayeh, S.A., E.T. Yu, and D. Wang, *III− V nanowire growth mechanism: V/III ratio and temperature effects.* Nano letters, 2007. **7**(8): p. 2486-2490.
9. Li, Z., et al., *Effects of nanowire length and surface roughness on the electrochemical sensor properties of Nafion-free, vertically aligned Pt nanowire array electrodes.* Sensors, 2015. **15**(9): p. 22473-22489.
10. Šupicová, M., et al., *Influence of boric acid on the electrochemical deposition of Ni.* Journal of Solid State Electrochemistry, 2006. **10**(2): p. 61-68.
11. Vicenzo, A. and P. Cavallotti, *Structure and electrokinetic study of nickel electrodeposition.* Russian Journal of Electrochemistry, 2008. **44**(6): p. 716-727.
12. Tsuru, Y., M. Nomura, and F. Foulkes, *Effects of boric acid on hydrogen evolution and internal stress in films deposited from a nickel sulfamate bath.* Journal of Applied Electrochemistry, 2002. **32**(6): p. 629-634.