



Template Synthesis of Ni Nanowires: Characterization and Modelling

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ABSTRACT

Template-assisted electrochemical deposition is a straight forward approach for the synthesis of 1D nanostructures (e.g., nanowire, nanorod, and nanobelt) with controllable morphology. This approach is suitable for mass production as it works at ambient pressure and temperature with the properties of synthesized 1D nanostructures being influenced by synthesis conditions during the electrochemical deposition process. This work aims to investigate the influence of stabilizing agent concentration and heating temperature towards the physical behavior of Nickel (Ni) nanowires synthesized via a template-assisted electrochemical deposition approach. In this research, the electrolyte bath was prepared in three different concentrations of the stabilizing agent (6 g/L, 40 g/L and 70 g/L), and the deposition bath temperature used was 30°C, 70°C, and 110°C respectively. The elemental composition was determined using Energy Dispersive X-ray (EDX) analysis to investigate the percentage of pure Ni element in the synthesized nanowires. The diameter, surface texture, and growth length of the synthesized Ni nanowires were characterized using Field Emission Scanning Electron Microscope (FESEM). X-ray diffractions (XRD) was used for crystal size and crystal orientation analysis. Additionally, the mechanical properties of Ni nanowires were extracted via molecular dynamic simulation. Growth length of Ni nanowires found to be significantly improved as the heating temperature increased, but it decreased when stabilizer agent concentration is high. The diffraction patterns for all synthesis conditions exhibited the synthesis Ni nanowires are polycrystalline as the crystalline planes with Miller indices of 111, 200, and 220. All the investigated nanowires showed ductile failure behavior, a typical behavior at larger length scales of Ni.

Keywords:

Nickel Nanowires; Template-Assisted Electrodeposition; Mechanical Properties

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1. Introduction

Nanotechnology scientists have developed numerous techniques to produce nanomaterials with different morphologies and configurations, which has led to interesting properties and behavior [1, 2]. Among the developed technique, the template-based approach perceived to be the most popular due to the ability to synthesize 1D nanostructures with controllable size and shape [3, 4]. This method is known for its simplicity, high-throughput, cost-effectiveness, and possible the duplication of complex topology present on the surface a template in a single step [5, 6]. The method typically operates at ambient temperature and pressure, as well as can be used for mass production of nanowires with controlled geometry and morphology [7-10]. For example, the diameters of nanowires can be well determined and maintained by controlling the diameter of pores, where else, the lengths can be controlled by controlling the thickness of the template [11]. This technique can be operated together with various types of templates with different shapes of 1D nanostructures for various materials [12, 13].

Among the available templates, anodic alumina oxide (AAO) templates are the most widely used as these templates have shown to be the most efficient medium for the synthesis of metallic nanowires. These templates have the advantage to control the diameter of nanowires precisely, while the diameter is dictated by the pore size of the nanochannels of AAO templates [7, 14-19]. Usually, the commercial template is used to avoid challenging template fabrication steps and focus on the nanowire synthesis parameters [20]. Apart from the AAO templates, other available templates are polycarbonate [21, 22], nanochannel glass [23], porous silica [24], mica film [25], and block copolymers [26].

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 numbers in the porous part of the alumina afford energetically favorable sites for initiating metal adsorption during electrodeposition [27]. The metal ion Mn^{+} 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 is schematically illustrated in Figure 1(a). Miguel García *et al.*, [28] 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 1(b). These are straightforward routes, but it is critical to ensure that the pores are filled with 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.

Blagg *et al.*, [27] synthesized niobium nanowires via template-based electrodeposition techniques by using ionic liquid as electrolyte. The synthesized nanowires are polycrystalline with grain size in the range of 14nm. Zhang *et al.*, [28] successfully synthesized Cobalt nanowires with a diameter size of 25 nm to 75 nm by varying the voltages supply to the electrochemical cell setup. The study revealed that high voltage supply would increase the growth rate of nanowires inside the template pores. Meier *et al.*, [29] used the same strategy for the fabrication of Copper nanowires and Nickel nanowires. Free-standing Copper nanowires and Nickel nanowires with a diameter size of 300nm and length size of 10 μ m were successfully synthesized by using AAO as a template.

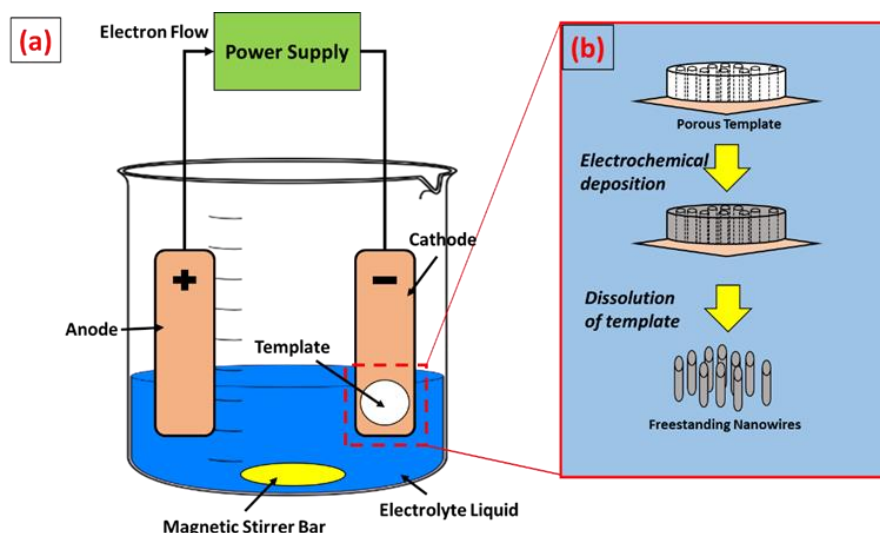


Fig. 1. (a) electrodeposition process (b) Nanowires formation process

The mechanical properties of nanostructures can be expected to change when the dimensions of crystalline material decreased to nano-size [29] due to the large surface-to-volume ratio, thereby resulting in different surface effects [30, 31]. Additionally, the gradual change of engineering product dimensions to nano-size has resulted in the complexation of the micro testing procedure, and the mechanical analysis procedure of individual nanostructures has become a significant challenge [32-34]. These are due to the extremely small nanostructures, prohibiting the applications of the standard process of mechanical testing methods. As such, to overcome these challenges and to provide further insight into the mechanical deformation of nanostructures, a numerical method such as molecular dynamics (MD) can be performed [35]. MD simulation is a statistical mechanics approach that is used by the material scientist to observe the atom movement during the application of load (e.g., bending, compression and tension) to extract this material behavior [36].

In this research, 1D Ni nanowires are synthesized via a template-assisted electrodeposition approach at different deposition temperatures (30, 70, and 110°C) and stabilizer concentrations (6, 40, and 70g/L). Then, the Ni nanowires are characterized to determine the synthesis condition effect towards the nanowires' structural properties. The elaboration of the physical properties of obtained Ni nanowires by considering the effect of stabilizer concentration and deposition bath temperature on the elemental composition, surface morphology, growth length, crystal orientation and crystal size of the synthesized Ni nanowires is presented in this article. The selection of the parameters in this research greatly dependent on the current research limitation where the investigation on the physical properties of metallic nanowires towards stabilizer concentration has not been studied and temperature being the most influential parameter in the synthesis process been opted for this research to understand the physical properties and the correlation. Lastly, the mechanical behavior of Ni nanowires is estimated by using the MD-based LAMMPS simulation method.

2. Methodology

2.1 Materials

The chemicals such as Nickel (II) chloride 6-hydrate (Bendosen,99.5%), Boric acid (Bendosen), and Sodium Hydroxide (Emsure ISO) were used without any further purification. Nickel plate with a thickness of 1.0 mm (99.95%) was procured from Aldrich Chemistry. Deionized water was used throughout the entire study.

2.2 Experimental Methods

The main content of the electrolyte bath for the electrochemical deposition procedure consists of metal salts and catalysts or stabilizers to accelerate the formation of nanowires. From the literature survey, experimental procedures done by Ertan *et al.*, [22] was followed due to the simple method with excellent outcomes. 175g/L of Ni sulfate hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$) metal salt solution was prepared and mixed with 6, 40, 70g/L of boric acid (H_3BO_3) solution. As explained in introduction part, both independent design variables, namely the solution bath temperature and concentration of boric acid, were varied while other design variables were kept constant. Research work carried out by Ertan *et al.*, [22] was used as a guidance to set boric acid concentration at 40 g/L and bath temperature at ambient condition (30 °C) as the baseline for this present study. The solutions then stirred until both chemicals well mixed. Figure 2 shows the apparatus arrangement for the electrochemical deposition process of Ni nanowires. Both Ni plate (anode) and ~200 nm pore size Whatman anodic alumina oxide (AAO) (cathode) dipped into an electrolyte (refer Figure 3). Figure 4 presents FESEM images of AAO templates with well-ordered nanopore arrangement with pore diameter in range of 200-250 nm. Circles represents nanopore with 200 nm diameter. Table 1 tabulated the parameters used for this experiment. The etching is the last procedure in the process of Ni nanowires synthesizing. Etching procedure involves dissolving of AAO templates by using 10g NaOH in 15ml distilled water to obtain free-standing deposited nanowires. The obtained free-standing Ni nanowires are then ready for analysis.

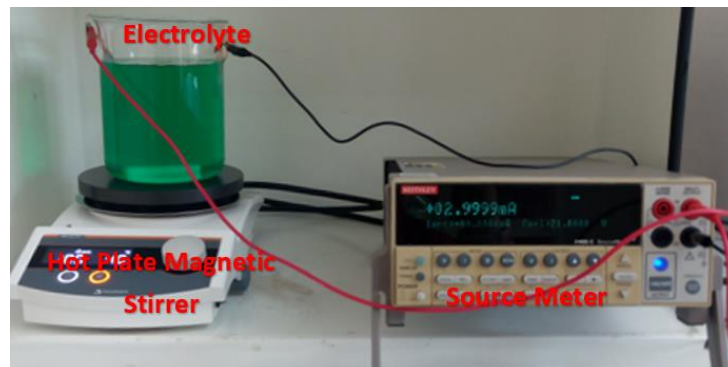


Fig. 2. Apparatus and equipment arrangement for cell setup

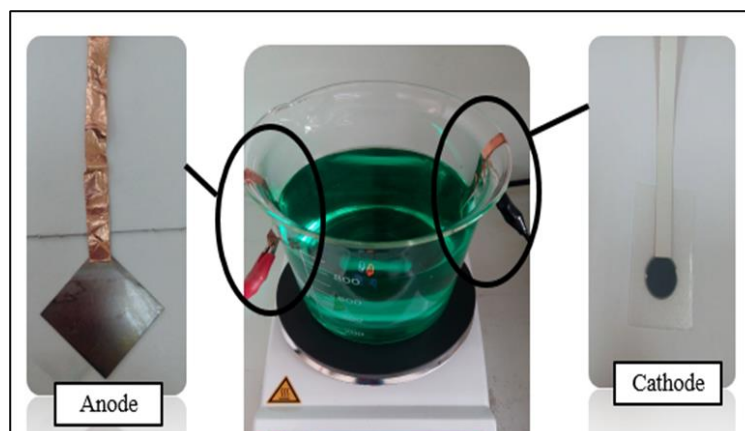


Fig. 3. Nickel plate as anode and AAO template as cathode

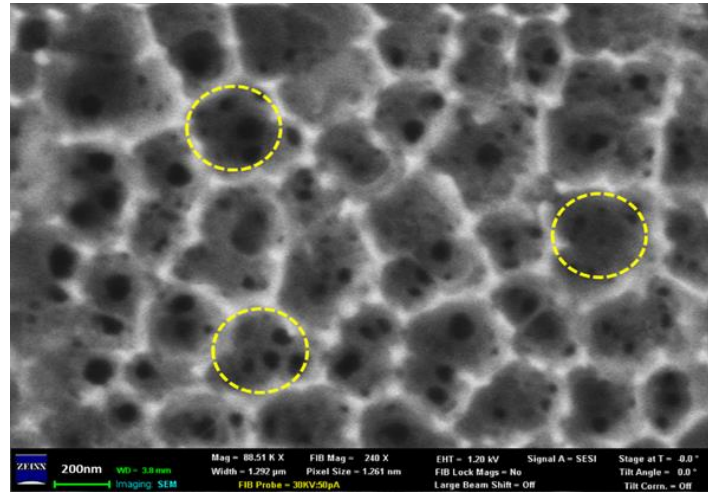


Fig. 4. FESEM image of AAO templates

Table 1

Synthesis parameter for electrochemical deposition

| Electrolyte | Stabilizer Agent Concentration (g/L) | Deposition Period (min) | Applied Current (mA) | Deposition Bath Temperature (°C) |
|-------------|--------------------------------------|-------------------------|----------------------|----------------------------------|
| I | 6 | 60 | 3.0 | 30 |
| | | | | 70 |
| | | | | 110 |
| II | 40 | 60 | 3.0 | 30 |
| | | | | 70 |
| | | | | 110 |
| III | 70 | 60 | 3.0 | 30 |
| | | | | 70 |
| | | | | 110 |

2.3 Characterization Techniques

Bruker D8 Advance X-ray diffraction (XRD) was used with radiation source operated at 40kV and 40mA to study the crystallographic properties of Ni nanowires. The EDX analysis is performed using Oxford Instruments EDX attached to JOEL JSM-7800F FESEM at a beam source of 15kV to elementary composition analysis and FESEM to investigate the morphology of the nanowire.

2.4 Molecular Dynamic Simulation

MD simulation was performed via Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS) software. LAMMPS software was developed by Sandia National Laboratories and usually used to model particles in various states with different base material by generating coding in the C++ language. This software has no numbers of atom limitations and able to perform simulation using different force fields and boundary conditions.

3. Results and Discussion

From the FESEM analysis, it was shown that well-defined and excellent repeatability of Ni nanowires synthesized at electrolyte temperature of 30 °C and boric acid concentration 40 g/L were generated inside the pores of AAO templates on the AAO templates with a uniform diameter (200 nm - 330 nm) as shown in Figure 5.

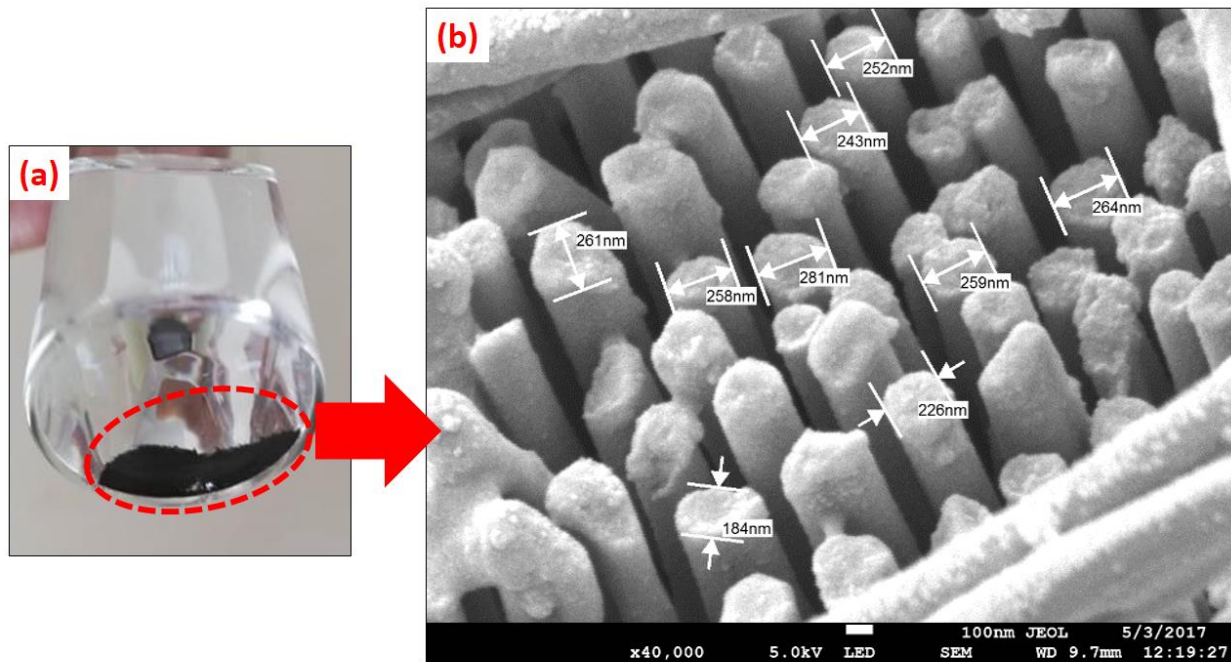


Fig. 5. (a) Etched Ni nanowires (b) Diameter measurement of synthesized Ni nanowires by FESEM

Elementary Composition -The EDX characterization proves that the Ni nanowires synthesized at electrolyte temperature of 30 °C and boric acid concentration 40 g/L were composed of 96.99% of Ni and 3.01% of O₂, as shown in Figure 6(a) and Figure 6(b). The small percentage of O₂ observed indicates the possible absorption of O₂ from the air on the surface of Ni nanowires.

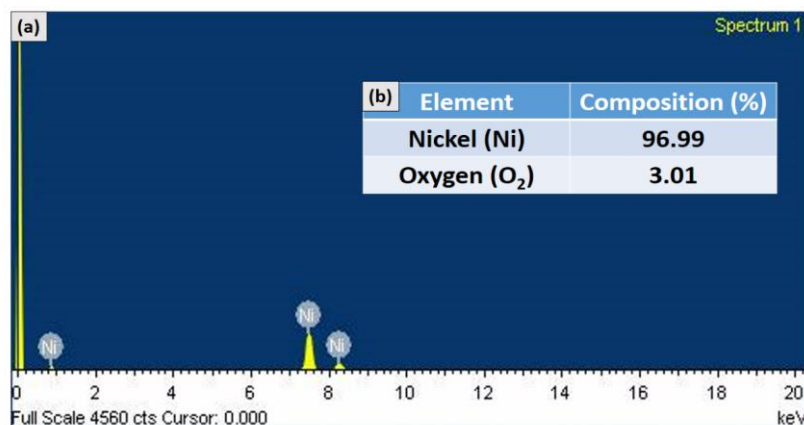


Fig. 6. EDX spectrum of synthesized Ni nanowires

Surface texture - From the FESEM analysis, it is noticed that all the synthesized Ni nanowires have a rough and flaky surface texture. Also, the observation found that there were significant differences

when the parameter of the synthesis condition changed. All the surface texture becomes rougher with the increasing of stabilizer agent concentration. This is due to the increasing of internal stress, which causes the cracks on the nanowires surface texture. This possible reason can be supported by Davolas *et al.*, [36] research findings; the presence of boric acid in the electrochemical deposition process causes the surface texture of deposited Ni to be glassy, instead of brittle and irregular surface appearance. Boric acid serves as buffering agents for electrochemical deposition [37], and it is crucial for hastening the formation of nanowires. However, the present research findings found to be opposite with the literature and the correlation of stabilizer agent concentration to the surface texture of Ni nanowires remains complex and ill-defined.

The surface texture also seen 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 of this phenomenon, large Ni grains is obstructed to generate inside the templates pore during the formation of nanowires [38], as shown in Figure 7.

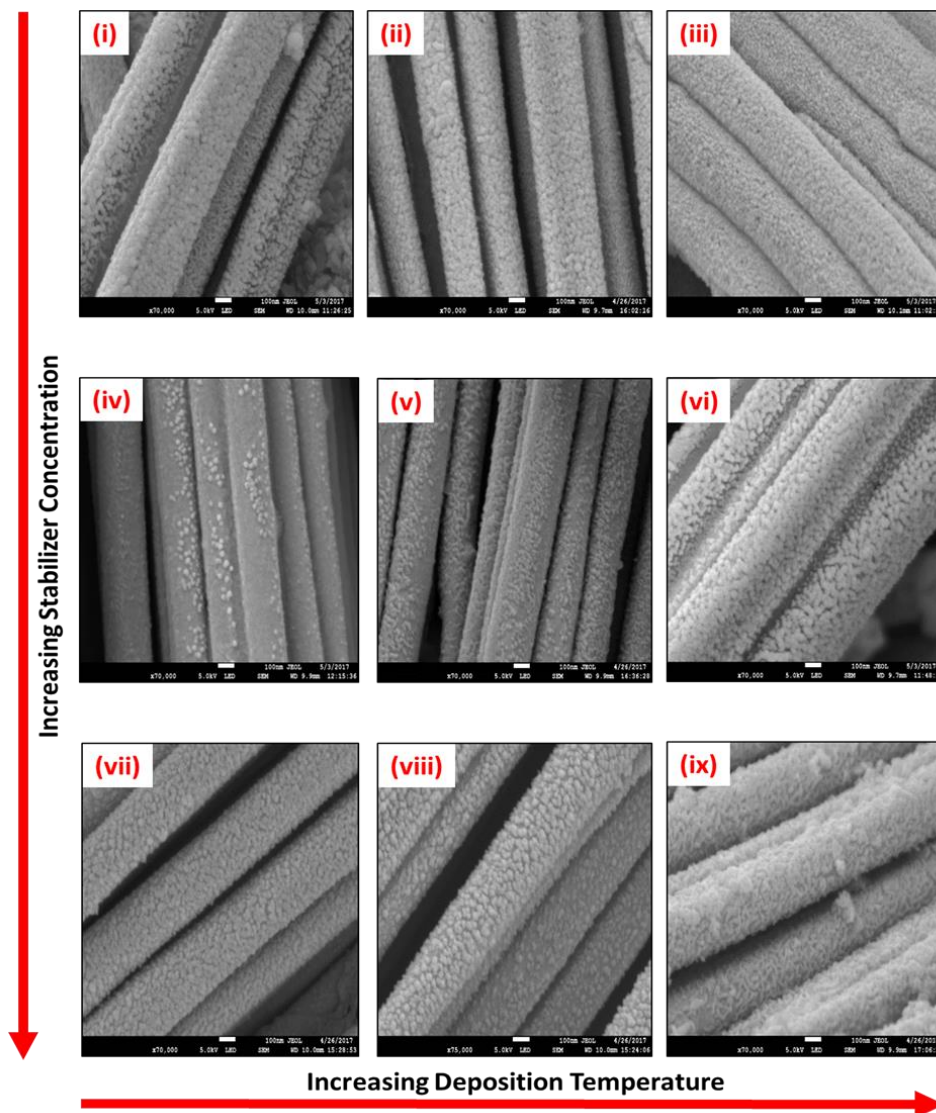


Fig. 7. FESEM analysis of synthesized Ni nanowires. (i-iii) stabilizer agents concentration of 6g/L and deposition temperature of 30°C, 70°C and 110°C respectively (iv-vi) stabilizer agent concentration of 40g/L and deposition temperature of 30°C, 70°C and 110°C respectively (vii-ix) stabilizer agent concentration of 70g/L and deposition temperature of 30°C, 70°C and 110°C

Growth length - Figure 8 shows the average measured growth length of the synthesized Ni nanowires for all parameters. The length of Ni nanowires found to be longer when the heating temperature set to be high. Higher temperature increases the rate of atomic diffusion [39], increases the speed of Ni nanowires formation, and thus accelerates the growth relative to the nucleation of new grains [40]. The influence of deposition temperature on the growth length of Ni nanowires was also due to the lowered energy barriers [41]. However, longer nanowire growth will create a higher center of gravity, which makes their position unbalanced [42].

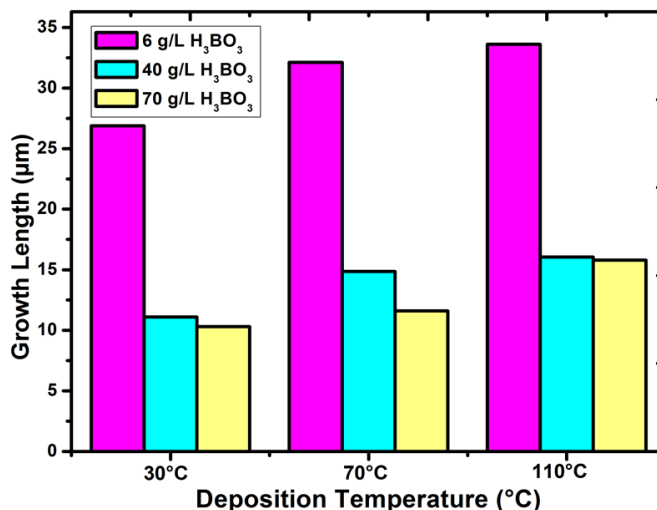


Fig. 8. Ni nanowire growth length for different synthesis conditions

Crystal Orientation - Figure 9 shows the XRD spectrum for the synthesized Ni nanowires at all synthesis conditions. The diffraction patterns for all synthesis conditions 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 increases as the deposition temperature increases showing that the quality of crystalline was increased with the higher deposition temperature [43].

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 gives the best crystal quality to the synthesized Ni nanowires. It can be concluded that stabilizer agent concentration influences the crystal quality of Ni nanowires with the ideal stabilizer agent concentration of 40g/L with a heating temperature of 110°C.

Crystal size - Figure 10 shows the calculated crystal size of Ni nanowires synthesized at all synthesis conditions. The results strongly suggest that the crystal size improves as the heating temperature set to be higher. This indicates that higher temperatures would exhibit the crystalline structure of the Ni nanowires due to the favoring growth of pre-existing nuclei during the initial stages of the electrochemical mechanism [39]. Deliberating the influence of stabilizer agent concentration to the crystal size of nanowires, the crystal size of synthesized Ni nanowires found to be slightly decreased as the stabilizer agent becomes more concentrated. Further analysis is needed to figure out the possible causes behind this hypothesis. Vincenzo and Cavallotti [44] revealed that when boric acid added to the electrolyte at pH above 4, they extended the stability field of the crystallographic and improved its quality of crystals produced. Another literature claimed that the reduction of boric acid concentration caused an increase in pH value, thus decreased the current efficiency (100% to 63%) [44].

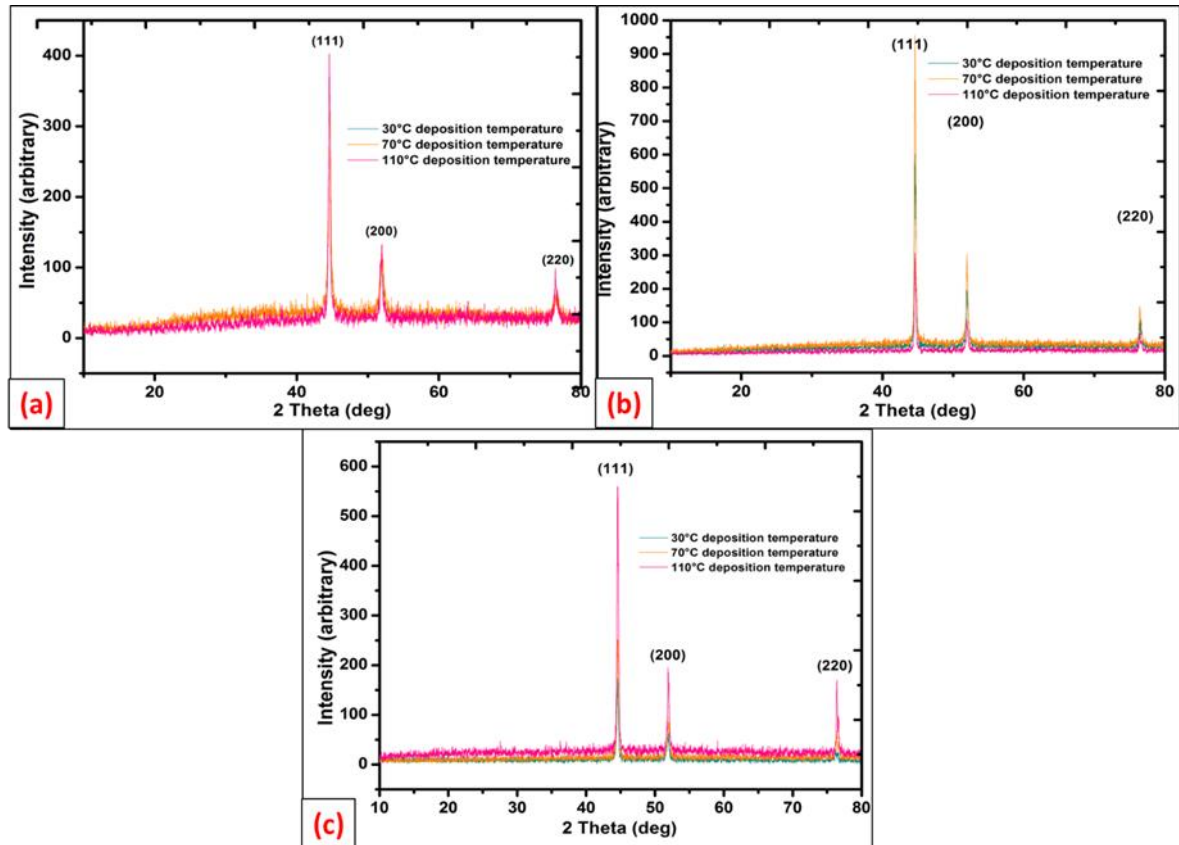


Fig. 9. XRD characterization. (a) 6g/L stabilizer agent concentration with a heating temperature of 30°C, 70°C and 110°C respectively (b) 40g/L boric acid concentration with a heating temperature of 30°C, 70°C and 110°C respectively (c) 70g/L boric acid concentration with deposition temperature of 30°C, 70°C and 110°C

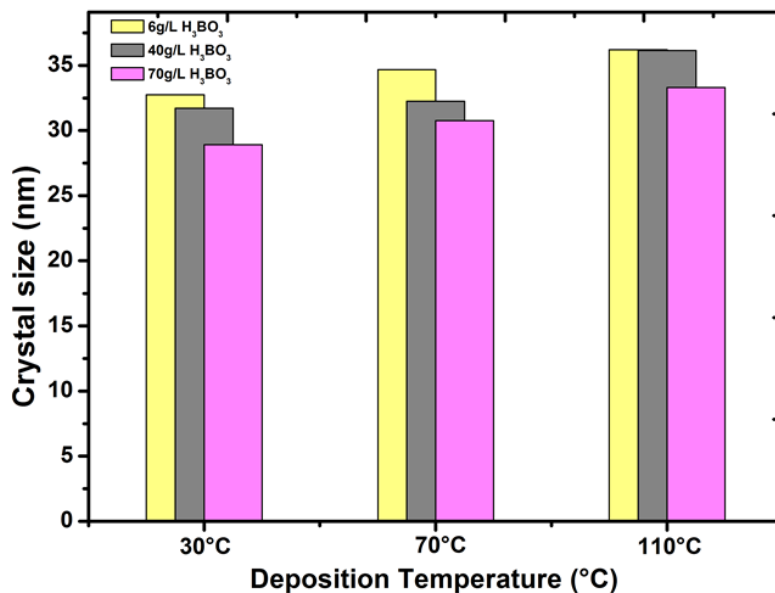


Fig. 10. Crystal size of Ni nanowires synthesized at different stabilizer agent concentration and heating temperature

3.4 Mechanical Properties of Ni Nanowires by MD Simulation

The parameter used in modeling Ni nanowires via MD simulation can be summarized as follows

- i. Force field : Embedded-atom method potential for Ni-Al alloys by Mishin
- ii. Temperature : 300 K
- iii. Timestep : 0.001
- iv. Relaxation period : 5000 steps
- v. Deformation : Tensile at X-direction at 0.0001 ps^{-1}

Table 2 tabulated Ni nanowire model dimensions. The diameter of the modeled nanowire was set to a smaller value compared to the actual value obtained from the experiment in order to reduce the simulation duration. Ni nanowires with various length sizes were simulated to estimate the influence of length size to mechanical behaviour of Ni nanowires. Figure 11 demonstrates the deformation process of model VI. During the relaxation state, some of the Ni atoms start to rearrange themselves, and the local lattice starts to defect [45]. After 5000 steps of the relaxation period, during the deformation stage, the surface of the nanowire is cracked abruptly, and some lattices slide on the cracking area resulting in the necking shape until it deforms into the final rupture. With the application of strain, the cyclical plastic deformation led to necking in the system, which culminates with the failure of the nanowire [46].

Table 2
 Nickel Nanowire Model Dimensions

| Model | Diameter (nm) | Length (nm) | Number of atoms |
|-------|---------------|-------------|-----------------|
| I | 8.05 | 18.5 | 62,828 |
| II | 8.05 | 22.11 | 75,398 |
| III | 8.05 | 24.63 | 87,964 |
| IV | 8.05 | 29.15 | 100,528 |
| V | 8.05 | 32.67 | 113,096 |
| VI | 8.05 | 36.1 | 125,660 |
| VII | 8.05 | 53.7 | 188,492 |
| VIII | 8.05 | 71.3 | 251,328 |

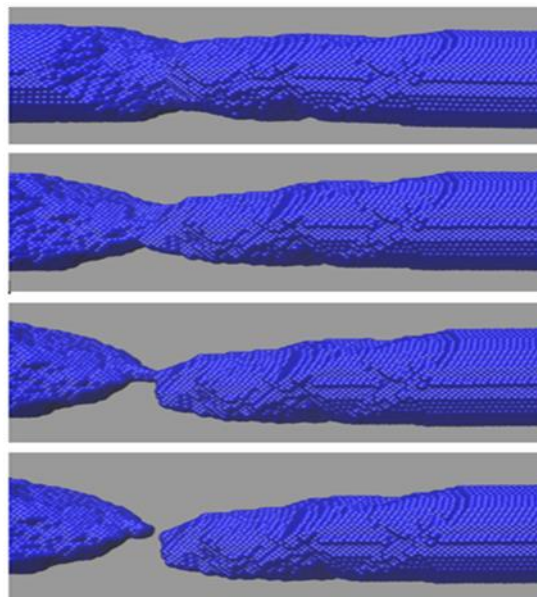


Fig. 11. Failure under the tensile deformation process of Ni nanowire

Figure 12 shows the stress-strain curve of all the modeled Ni nanowires. The stress-strain curve for all Ni nanowires models are entirely overlapped with the stress values are increasing up to approximately 16.4 GPa, and the strain shows that the length size has no significant influence on the mechanical properties of Ni nanowires. The yielding stress obtained in this study found to agree with the findings from Wang *et al.*, [47], and Mohan *et al.*, [48] reported the yielding stress of 14.8 GPa and 15-17 GPa, respectively.

Table 3 tabulated the obtained tensile modulus from this simulation is in the range of 140.03-142.60 GPa, which is about 70% of its bulk value. Where else, Wang *et al.*, [47] reported that the tensile modulus in the range of 271 GPa - 324 GPa from the MD simulation of different sizes of Ni nanowires, while Dupont *et al.*, [49] reported slightly similar finding which is equal to 291 GPa. Additionally, Huang *et al.*, [33] in counterpart reported a higher value of tensile modulus, which is in the range of 366-611 GPa. However, Wang *et al.*, [47] and Mohan *et al.*, [48] reported lower value modulus, which is about 134 GPa and 182.71 GPa, respectively. The variance in the obtained result can be ascribed to the periodic boundary condition and force field used for the modeling.

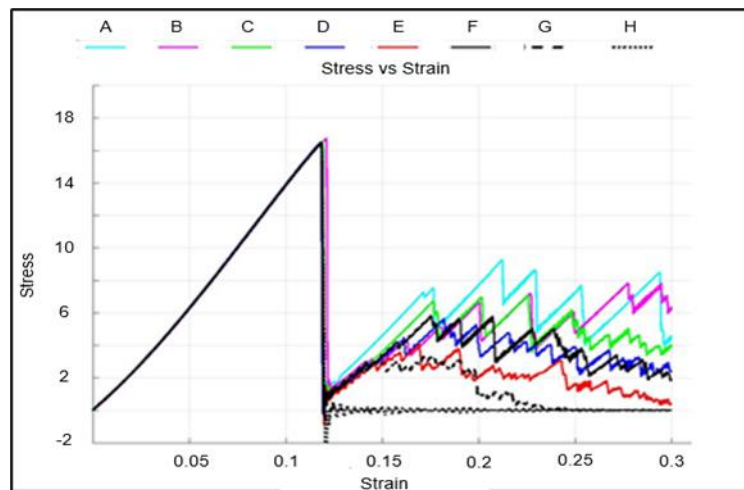


Fig. 12. Stress-strain curve of modeled Ni nanowire

Table 3

Simulated Mechanical Properties of Nickel Nanowires

| Model | Young Modulus (GPa) | Yielding Stress (GPa) | Yielding Strain (GPa) | Failure mode |
|-------|---------------------|-----------------------|-----------------------|--------------|
| I | 140.03 | 16.520 | 0.1181 | Ductile |
| II | 140.43 | 16.742 | 0.1208 | Ductile |
| III | 142.40 | 16.578 | 0.1189 | Ductile |
| IV | 142.60 | 16.537 | 0.1181 | Ductile |
| V | 140.44 | 16.481 | 0.1186 | Ductile |
| VI | 140.69 | 16.494 | 0.1180 | Ductile |
| VII | 140.60 | 16.475 | 0.1184 | Ductile |
| VIII | 140.54 | 16.476 | 0.1184 | Ductile |

4. Conclusions

Well-defined and excellent repeatability of Ni nanowires was successfully deposited on the AAO templates with a uniform diameter in the range of 200-330nm via a template-assisted electrochemical deposition approach. The obtained Ni nanowires with high purity (97.97% of Nickel from the EDX result) have a rough and flaky surface texture for all the synthesis condition cases. Observation from FESEM analysis also claims 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 significantly improved as the heating temperature increased, but it decreased when stabilizer agent concentration is high. The diffraction patterns for all synthesis conditions 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 increased as the deposition temperature increased, showing that the quality of crystalline was improved with the higher deposition temperature. It is also noted that the average crystal size increased when stabilizer agent concentration decreased. Where else, the crystal size increased when the heating temperature increased. Further investigation is needed to fully understand the rational reason for the characterization of the obtained Ni nanowires influenced by the stabilizer agent concentration and heating temperature. Eight Ni nanowires models were successfully developed and modeled by using LAMMPS simulation software. The finding showed that the modeled Ni nanowires have a tensile modulus between 140.03-142.60GPa, yielding stress between 16.475 to 16.742GPa, and yielding strain between 0.1180 to 0.1208. All the investigated nanowires showed ductile failure behavior, a typical behavior at larger length scales of Ni.

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