Type of contribution:
Editorial
▶ Research Paper
Case Study
Review Paper
Scientific Data
Report of Tech. Application

mesi
e-issn. 2798-5245

Mechanical Engineering for Society and Industry
Vol. 3, No. 2 (2023) pp 105-113
https://doi.org/10.31603/mesi.10493

Effect of Current, Time, Ethanol Concentration, and pH Electrolyte on ZnO Coated Carbon Fiber Using Electrochemical Deposition Method

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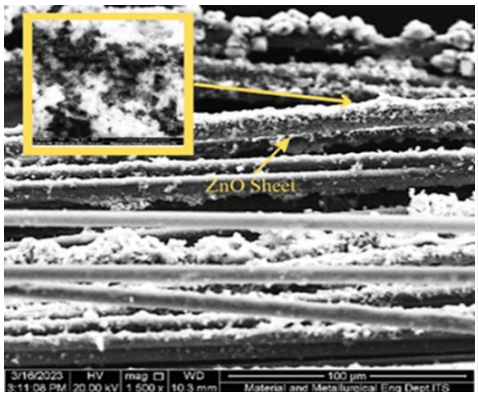
This article contributes to:



Highlights:

- This study explores the use of ZnO as a coating material on carbon fiber to create piezoelectric materials.
- A new approach involving current, time, ethanol concentration, and electrolyte pH was studied.
- Certain parameters, such as an electrolyte current of 1.4 A, higher pH (6.0), 70% ethanol concentration, and longer coating duration (270 seconds), produce a thicker and more uniform layer and influence the crystal structure formed.

Abstract

One of the recent developments in carbon fiber is using nano zinc oxide (ZnO) as a coating on carbon fiber to create piezoelectric materials. Piezoelectric materials can generate electricity when subjected to mechanical pressure or strain, and vice versa. ZnO nanomaterials have been a focal point of research due to their high surface-to-volume ratio and high reactivity. This study reported on the use of ZnO for coating agents in carbon fiber sensors. The novelty in this research is the composition of current, time, ethanol concentration, and pH electrolyte to produce the optimum composition of piezoelectric material. The process was conducted using an electrochemical method, which converts electrical energy into chemical energy through electro-deposition. This study considers four independent variables: electrolyte current (1.2 A and 1.4 A), electrolyte pH (2.0, 4.0, and 6.0), ethanol concentration (70% and 96%), and coating duration (90, 180, and 270 seconds). The results show that 1.4 A produces the highest average voltage, followed by electrolyte pH 6 and 70% ethanol concentration. The best coating time was 270 seconds producing the highest average voltage. Micro and SEM confirm that 1.4 A produced a thicker and more uniform layer compared to 1.2 A. High pH, 70% ethanol concentration, and longer coating time also contributed to the formation of thicker layers. XRD test shows that the layers formed had amorphous and hexagonal crystal structures. The average crystal diameter size varies depending on the combination of independent variables used in the coating process. With these results, piezoelectric has potential as a pulse sensor material.

Keywords: Piezoelectric, Carbon fiber, ZnO, Electrochemical deposition

Article info
Submitted: 2023-11-05
Revised: 2023-11-28
Accepted: 2023-12-01

Publisher
Universitas Muhammadiyah Magelang

1. Introduction

Carbon fibers (CF) as reinforcement for polymer matrix composites began to be used for commercial production in the 1960s. Carbon fibers are used in a variety of potential applications, especially in the fields of mechanics, aerospace, and industrial engineering [1]. Carbon fiber has excellent tensile properties, low density, high thermal and chemical stability without oxidizing, good thermal and electrical conductivity, and excellent creep resistance [2]. Combining carbon fiber with other chemicals makes its development very fast. One of them is coating carbon fiber using nano Zinc Oxide which can be used as Piezoelectric and nano generator [3].

Nanomaterials are widely developed because they are very useful in biomedical life and have unique properties of nanomaterials, such as high surface-to-volume ratio and high reactivity. ZnO as a semiconductor has a wide direct band gap (3.37 eV) with a large exciton binding energy (60 meV) making it suitable for use in the optoelectronic, photocatalyst, and piezoelectric fields [4]–[7]. Nano ZnO has the advantages of biocompatibility, biodegradability, and low toxicity, making it very suitable for applications in the analytical field [4], [8]. ZnO nanostructures possess outstanding features (wide band gap, large excitation binding energy, non-toxicity, biocompatibility, chemical, and photochemical stability), making them suitable compounds for producing biosensors, gas sensors, solar cells, and nanogenerators [5], [9]–[13].

There are many methods of coating substrates in the manufacture of biosensors such as piezoelectric, including Atomic Layer Deposition (ALD) [10], Chemical Vapor Deposition (CVD) [9], Electrochemical Deposition, Sputtering, and Pulsed Laser Deposition [11]–[17]. One of the recommended methods is the electrochemical method [6], [18]. Electrochemical are one way to coat carbon fiber. Electrochemical is a reaction process of exchanging electrons between two conductors, namely electronic conductors, and ionic conductors. Initially, electrochemical used solids as electrodes and liquids as coatings. However, over time it can use solids as a coating [7], [19]. In this research, carbon fiber coating with nano ZnO will be carried out using the electrochemical deposition method to make piezoelectric material as a pulse sensor material. This research was carried out with the variable concentration of ethanol as an electrolyte, coating reaction time, electrolyte pH, and the current used.

2. Methods

This research was carried out using three processes, namely the carbon fiber preparation process, electrolyte preparation, and the ZnO coating process onto the carbon fiber using the electrochemical deposition method.

2.1. Carbon Fiber Preparation

The materials used in this study were carbon fiber, zinc acetate dihydrate (ZAD), ethanol, HNO_3, and NH_4OH. Prior to coating the carbon fiber, it was treated first by immersing the carbon fiber in acetone for 72 hours, then soaking it in nitric acid for 72 hours, then cleaning it with distilled water and ethanol, then soaking it again in a solution of ZAD and ethanol for 2 hours. Then, it was dried at 100 °C. This process was carried out for seeding ZnO seeds.

2.2. Electrolyte Preparation

Electrolyte preparation was carried out by dissolving ZAD in ethanol with a ratio of 1 gram/50 ml. The electrolyte that has been mixed with ethanol is then added with HNO_3 and/or NH_4OH until it has a pH that corresponds to the desired pH, namely having a pH of 2.0; 4.0; and 6.0.

2.3. Coating Process

The coating process carried out in this study used the electrochemical method where the electrode material used a nickel plate with a size (50 mm x 20 mm x 0.2 mm). Then the electrolyte solution was electrified by 1.4 amperes for 90 seconds, 180 seconds, and 270 seconds. After the electrochemical coating process was completed, the carbon fiber was dried at room temperature for ± 24 hours. The scheme of electrochemical coating of carbon fiber is shown in **Figure 1**.

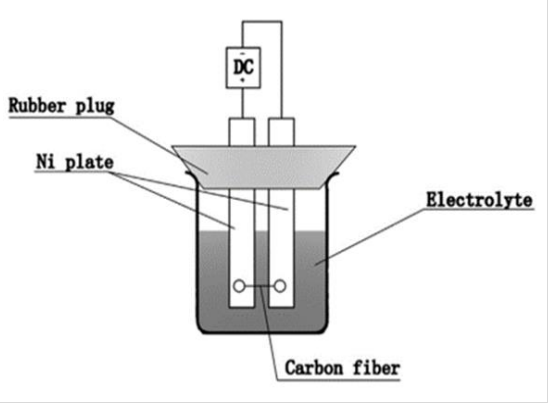


Figure 1. Schematic of electrochemical deposition [3]

2.4. Characterization

Specimens are prepared for piezoelectric performance testing. Then the specimen preparation was carried out, namely grinding, polishing, and etching using 5% HF solution. After that, the microstructure of the specimen was observed using an optical microscope (Olympus Microscope BX41M) and an electron microscope (SEM). In addition, XRD testing was also carried out.

3. Results and Discussion

3.1. Carbon Fiber Performance Test

In this research, performance testing was carried out with 4 variables namely, the effect of pH, current, electrolyte concentration, and coating duration. This test was carried out during the electrochemical deposition process using a current of 1.2 A and 1.4 A and an electrolyte solution having a pH of 2, 4, and 6 with a coating time of 90 seconds, 180 seconds, and 270 seconds. From the results of the study, data were obtained as shown in **Figure 2(a)** to **Figure 2(d)**.

Figure 2(a) illustrates that at pH 6, a voltage value of 0.2949 volts is generated. This value is higher compared to pH 2 and pH 4. **Figure 2(b)** explicates that with an increase in the applied current, the voltage value also increases, reaching the highest voltage at a current of 1.4 A, which is 0.2949 volts. **Figure 2(c)** can be elucidated by noting that a 70% ethanol concentration produces a higher voltage compared to a solution with 96% ethanol. **Figure 2(d)** shows that the coating time given will increase the generated voltage. From this research, the highest time is 270 seconds. This is the longer the time given the ZnO layer will be thicker so that it will increase the value of the tension generated by the carbon fiber. The thickness of the layer affects the stress that can be generated by the specimen, the thicker the layer, the higher the stress that can be generated by the specimen [8], [20]. In general, as the thickness of the ZnO layer on the carbon fiber increases, the ZnO surface area exposed to pressure increases, so that the response given by the piezoelectricity will be greater. This is the reason why the thicker the ZnO layer on the carbon fiber will increase the resulting stress [10], [21]. Similar research was conducted in the previous study, where Li and Wang studied that coating ZnO on a substrate (carbon fiber) is very useful for pulse sensor materials. Although the resulting output is relatively small, it can be used as a respiratory sensor in biomedical and healthcare applications [9], [11].

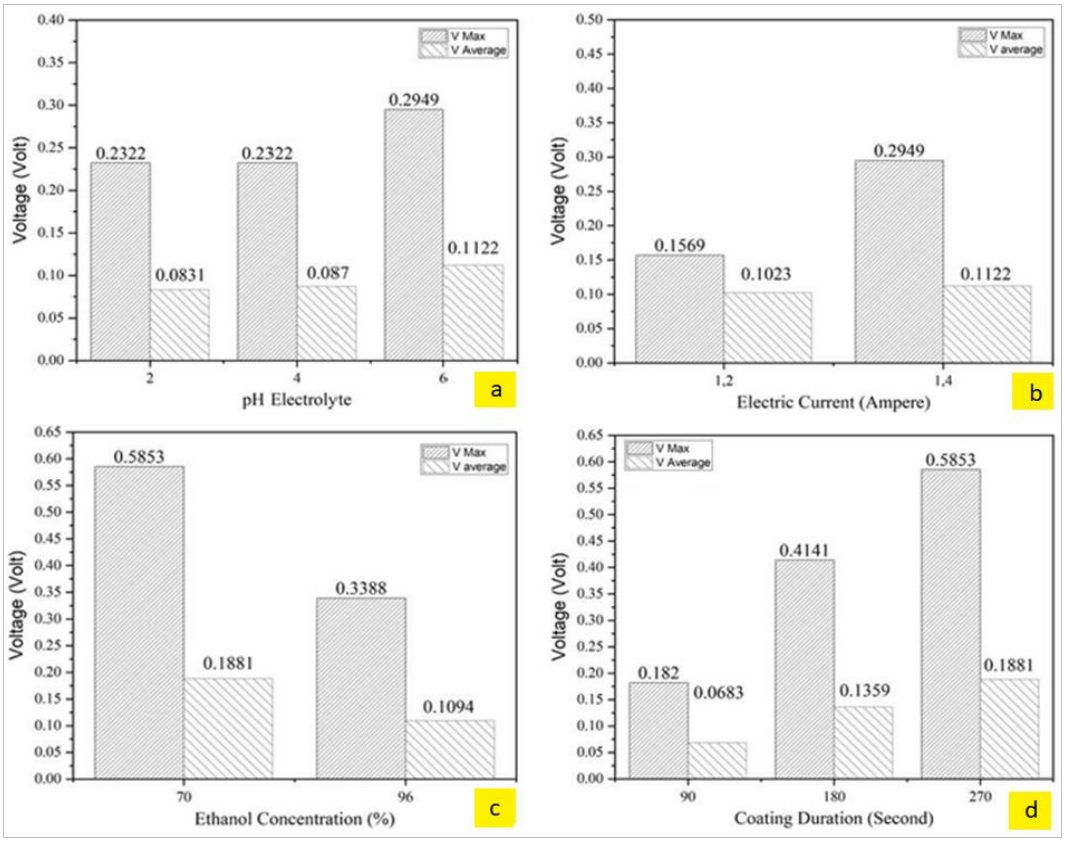


Figure 2. (a) The voltage with a current of 1.4 A in the electrochemical process with an electrolyte pH of 2.4 and 6; (b) The voltage with electrolyte pH 2 and current used for the electrochemical deposition process of 1.2 A and 1.4 A; (c) Stress with coating time of 270 seconds and ethanol concentrations used for the electrochemical deposition process of 70% and 96%; and (d) Stress with 70% ethanol concentration in the electrochemical process with coating times of 90 seconds, 180 seconds, and 270 seconds

3.2. Effect of Current Used on Microstructure of Carbon Fiber

Figure 3(a) and **Figure 3(b)** does not show a significant difference, but if observed the ZnO layer on the surface of the carbon fiber at a current of 1.4 A is thicker than the ZnO layer with a current of 1.2 A. The process of coating ZnO on the surface of carbon fiber using a higher current will result in ZnO crystals growing faster. This results in the greater current will result in a thicker layer produced, at the same time of coating [3].

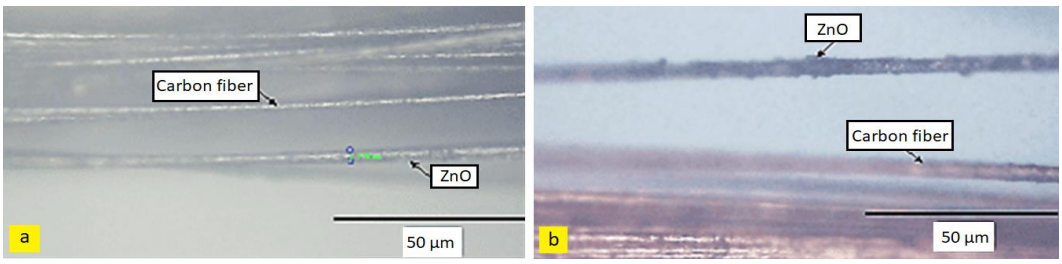


Figure 3. Carbon fiber with various currents during the electrochemical deposition process: (a) current 1.2 A, and (b) current 1.4 A

3.3. Effect of Current Used on Appearance of Carbon Fiber Micro

Figure 4(a) and **Figure 4(b)** shows that time has an important role in coating using the electrochemical method. This coating process uses electrochemical reactions, where the coating process takes time to reach the desired level of deposition or layer formation. The electrochemical process involves the isolation of electric charges between the electrodes and the electrolyte solution. In electrochemical plating, a coating of metal or other compounds usually occurs on the surface of the electrode. When the time for giving the electric current is extended, the ZnO particles attached to the substrate will get thicker [3].

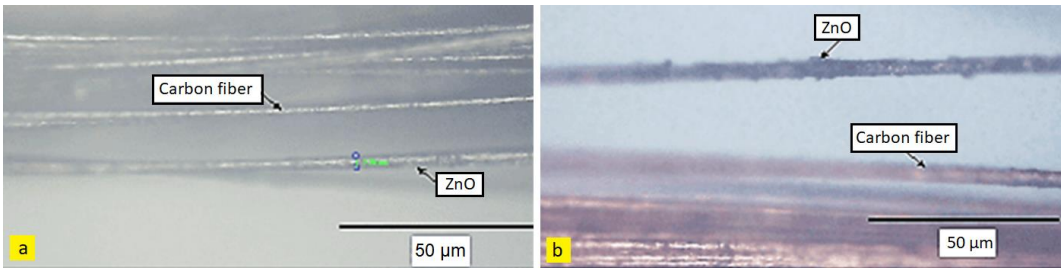


Figure 4. Carbon fiber with various currents during the electrochemical deposition process: (a) current 1.2 A, and (b) current 1.4 A

3.4. Effect of Coating Time on Carbon Fiber Microstructure

The concentration of ethanol as a solvent (electrolyte) with an electric current of 270 seconds and a current of 1.2 A shows a very significant change in the producing process of a thin layer on the surface of the carbon fiber, as can be seen from the comparison between **Figure 5(a)**, **Figure 5(b)** and **Figure 5(c)**. It can be observed that with a potential difference the level of ZnO coating on carbon fiber decreases. This can be related to the resistance of the electrolyte. When the concentration increases, the resistance decreases and this decreases the potential difference [11].

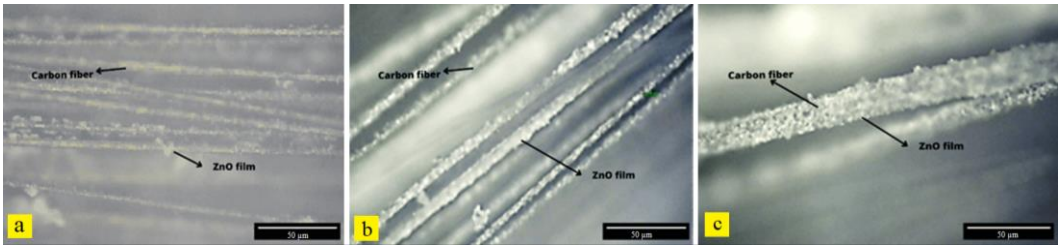


Figure 5. Coating of carbon fiber in the electrochemical deposition process with a time of: (a) 90 seconds; (b) 180 seconds; and (c) 270 seconds

3.5. Effect of Ethanol Concentration on Carbon Fiber Microstructure

Ethanol was used as a solvent (electrolyte) at 70% as seen in **Figure 6(a)** and ethanol at 98% as seen in **Figure 6(b)**. The time given is 270 seconds with an electric current of 1.2 A, which can provide a very significant change in producing a thin layer on the surface of carbon fiber.

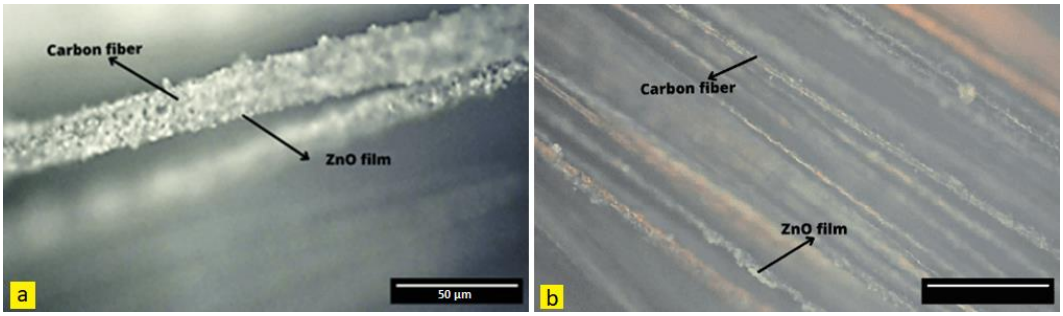


Figure 6. Carbon fiber based on ethanol concentration by electrochemical deposition method: (a) Ethanol 70%, and (b) Ethanol 96%

Carbon fibers with lower ethanol (70%) can produce thicker ZnO layers. According to Ref. [22], when the electrolyte concentration is increased, the amount of metal stored will decrease . It can also be observed that the potential difference also decreases, and this is related to the electrolyte resistance. As the concentration increases, the resistance decreases and this decreases the potential difference [23].

3.6. SEM Observations

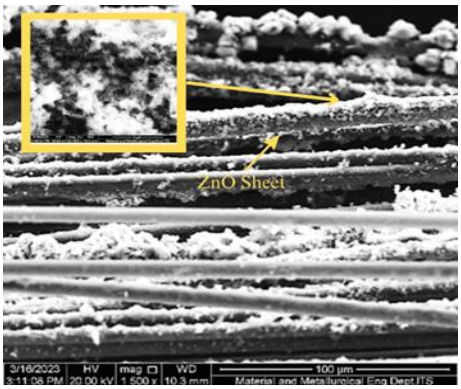


Figure 7. SEM image of the ZnO coating process with a current of 1.2 A and an electrolyte pH of 6

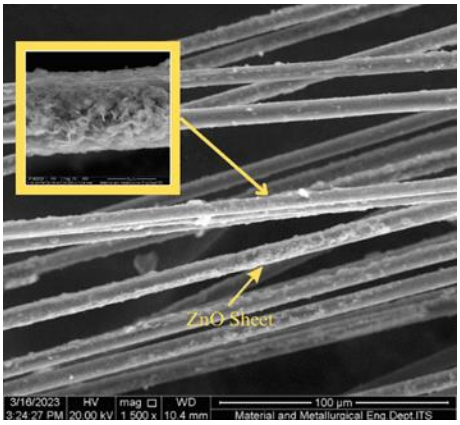


Figure 8. SEM image of the ZnO coating process with a current of 1.4 A and an electrolyte pH of 2

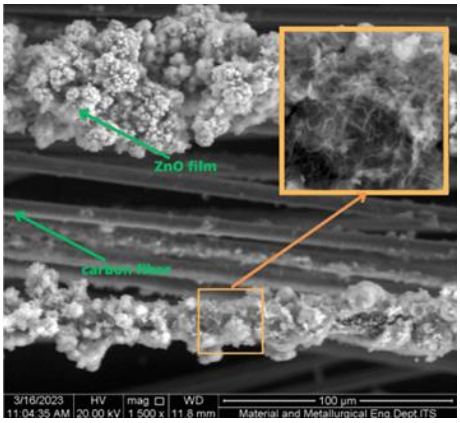


Figure 9. SEM image with 70% ethanol concentration and 270 seconds of coating time pH

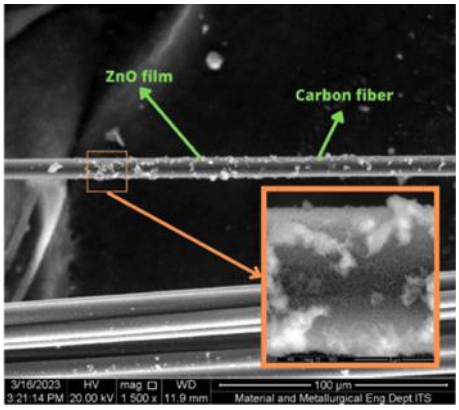


Figure 10. SEM image with 96% ethanol concentration and 90 second coating time

Figure 7 shows the ZnO layer (white layer) on the carbon fiber surface and **Figure 8** shows carbon fiber that is coated using variations in current strength during the electrochemical deposition process with a current of 1.2 amperes and the pH of the electrolyte used is 6. **Figure 8** depicts carbon fiber coated with ZnO using the electrochemical deposition method and using a current of 1.4 amperes with an electrolyte pH of 2. The difference that can be seen is the carbon fiber coated with ZnO at a current of 1.4 A and an electrolyte pH of 2 (**Figure 7**) appears to have better ZnO layer uniformity when compared to a current of 1.2 A and an electrolyte pH of 6 (**Figure 8**). The analysis of the results of this study was that all substrates were successfully coated with a layer of ZnO that looked like a white layer. From **Figure 8** and **Figure 9**, electrolyte pH 2 produces ZnO layers with a more uniform particle shape when compared to pH electrolyte 6.

Meanwhile, electrolyte pH 6 produces large hexagonal sheet-shaped layers that have non-uniform sizes. The lower the pH of the electrolyte used, the more uniform the layer is formed. This occurs because low pH has low availability of OH- ions. $Zn(OH)_2$ ions as the basic ingredient of ZnO require OH ions to react and support the formation of small and uniform spherical layer particles. Meanwhile, for electrolytes with a pH of 6, OH- ions are available in greater quantities. This supports the continuous growth of Zn(OH) at the cathode as the beginning of the formation of ZnO particles. This causes the coating particles to form larger sheets [13], [24].

From **Figure 7** to **Figure 10**, it can be observed that the ZnO layer formed is a nanosheet. ZnO nanosheet is a nanomaterial structure in the form of a thin sheet of zinc oxide. ZnO nanosheet has interesting properties because of its very small size and large surface area. This large surface area allows for stronger interaction with the surrounding environment, which can be useful in a variety of applications. ZnO is a semiconductor material that has antimicrobial properties due to its toxic effect on microorganisms [14], [25]. When ZnO is in the form of nanosheets, the large surface area allows for more intense interactions between ZnO and microorganisms. ZnO nanosheets can produce zinc ions (Zn2+) when exposed to water. These zinc ions can damage the cell walls of microorganisms, disrupt their metabolism, and cause cell damage which eventually results in the death of microorganisms.

3.7. XRD Test

Graphics of the XRD Origin test are shown in **Figure 11** and **Figure 12**. From the XRD data, the curve of the XRD test results has many wavy and wide peaks. **Figure 11(a)** shows a comparison of the curves of the XRD test results for specimens made with a current of 1.2 A and an electrolyte pH of 6 that matches the database serial number 96-900-4180. It states that the crystal system formed is hexagonal and has an average crystal size of 27. 16 nm. In **Figure 11(b)**, the curve with a current of 1.4 A and an electrolyte pH of 2 matches the database with serial number 96-230-0117. Where the material formed has a hexagonal crystal system and an average crystal size of 13.36 nm.

In **Figure 12(a)**, ZnO coating on carbon fiber with 70% ethanol concentration and a coating duration of 270 seconds matches the database with serial number 96-230-0115. It is stated that the crystal system formed is hexagonal and the average crystal size is 6.665 nm. In **Figure 12(b)**, the ZnO coating on carbon fiber with 96% ethanol concentration and coating duration of 90 seconds matches the database with serial number 96-230-0117. The material formed has a hexagonal crystal system and an average crystal size of 17.764 nm. The XRD curve produced by a crystal-shaped material is a curve that has a high-intensity peak, this is due to the scattering of X-rays in certain directions formed by atoms. Meanwhile, the XRD curve of an amorphous material does not have a high-intensity narrow peak because the X-rays are scattered in all directions [15], [26].

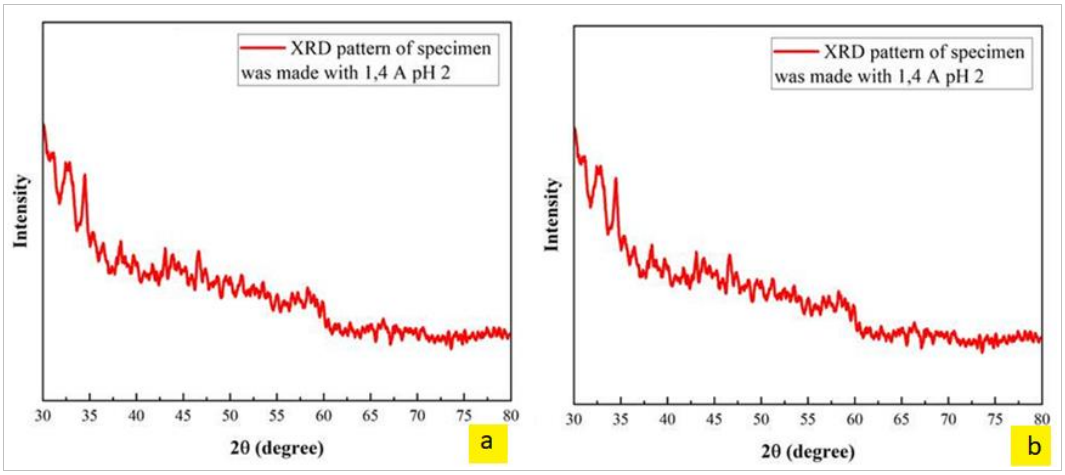


Figure 11. XRD test with variations in current and electrolyte pH using the electrochemical method: (a) 1.2 A, pH 6; and (b) 1.4 A, pH 2

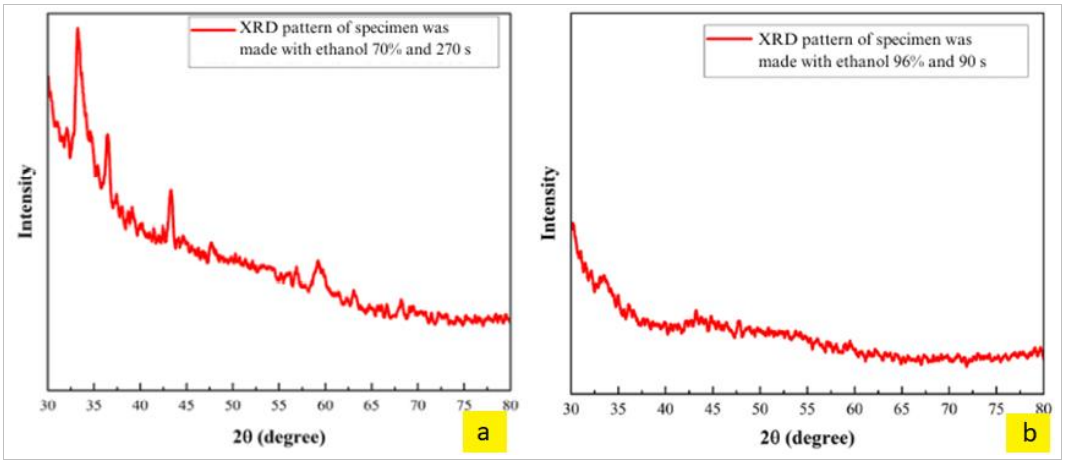


Figure 12. XRD test specimens with ethanol concentration and electrochemical coating time (a) 70%, 270 seconds and (b) 96%, 90 seconds

4. Conclusion

Sensor performance testing shows that a current of 1.4 A produces an average voltage of 0.1122 volts while a current of 1.2 A produces an average voltage of 0.1023 V. For research variables based on the influence of the pH of the electrolyte solution, data obtained with a pH of 2 produces the lowest average voltage of 0.0831 V while electrolyte pH 6 produces an average voltage of 0.1122 V. For the research variables based on the concentration of ethanol as the electrolyte, the best performance test results are obtained at an ethanol concentration of 70% with the highest voltage of 0.5853 V while at 96% ethanol concentration the resulting voltage was

0.3388 V. For research variables based on the length of coating time, the results of piezoelectric performance testing with a coating time of 270 seconds produced an average voltage of 0.1881 V.

The results of micro and SEM testing show that the results of ZnO coating using a current of 1.4 A have a layer of ZnO onto the surface of the carbon fiber that looks thicker and more even when compared to the current use of 1.2 A. Meanwhile, the coating results using a current of 1.2 A and pH 6 has a ZnO layer with a thickness of 2.871 µm. Meanwhile for ZnO coating using a current of 1.4 A and pH 2 has a ZnO layer with a thickness of 0.537 µm. The ZnO layer formed using a pH 2 electrolyte looks more uniform when compared to a pH 6 electrolyte solution. Coating ZnO using a 70% ethanol solution and a coating time of 270 seconds obtains a thicker and more even coating with a layer thickness of 10. 29µm. Coating ZnO using 96% ethanol solution with a coating time of 90 seconds has a ZnO layer thickness of 0.732 µm.

In the XRD test, it is known that the layer formed from this process has an amorphous shape and a hexagonal crystal system. For a ZnO coating current of 1.2 A with a pH of 6, it has an average crystal diameter of 27.162 nm. For a ZnO coating current of 1.4 A with a pH of 2, it has an average crystal diameter of 13.631 nm. ZnO coating using 70% ethanol solution and a coating time of 270 seconds has an average crystal diameter of 6.665 nm. For coating ZnO using a 96% solution and a coating time of 90 seconds has an average crystal diameter of 17.764 nm.

Acknowledgments

This research was funded and supported by University of Jember.

Authors' Declaration

Authors' contributions and responsibilities - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation, and discussion of results. The authors read and approved the final manuscript.

Funding –No funding information from the authors.

Availability of data and materials - All data are available from the authors.

Competing interests - The authors declare no competing interests.

Additional information – No additional information from the authors.

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