Characterization and Fabrication of Piezoelectric Energy Harvesting ZnO Nanorod on Textile by Hydrothermal Method

Muhammad Farhan Affendi Mohamad Yunos
Department of Electrical and Computer Engineering, Kulliyyah of Engineering, IIUM
Kuala Lumpur, Malaysia
farhanfendi93@gmail.com

Anis Nurashikin Nordin
Department of Electrical and Computer Engineering, Kulliyyah of Engineering, IIUM
Kuala Lumpur, Malaysia
anisnn@iium.edu.my

Aliza Aini Md Ralib
Department of Electrical and Computer Engineering, Kulliyyah of Engineering, IIUM
Kuala Lumpur, Malaysia
alizaaini@iium.edu.my

Zambri Samsudin
Jabil Circuit Sdn Bhd
Bayan Lepas Industrial Park Phase 4
Penang, Malaysia
zambrisamsudin@jabil.com

Lim Lai Ming
Jabil Circuit Sdn. Bhd.
Bayan Lepas Industrial Park Phase 4
Penang, Malaysia
laiming_lim@jabil.com

2023 IEEE International Conference on Sensors and Nanotechnology (SENNANO) | 979-8-3503-3331-2/23/$31.00 ©2023 IEEE | DOI: 10.1109/SENNANO57767.2023.10352533

***Abstract*— Recent advancements in sensing technology and wireless communications have accelerated the development of the Internet of Things (IoT) and wearable sensors. Piezoelectric energy harvesting device provides a promising solution for wearable energy harvester. Energy harvesting from wearable textiles can be achieved through various mechanisms, such as piezoelectricity, thermoelectricity, and photovoltaics. Zinc Oxide (ZnO) nanorods structure has garnered considerable interest among researchers in recent years due to its unique and favorable properties. This paper presents a fabrication process of a flexible piezoelectric energy harvester via hydrothermal method to produce ZnO nanorod structure. The electrode fabrication was discussed using screen printing method to grow the ZnO nanorods on top of the electrode surface. The characterization of the ZnO was done by SEM, XRD and piezometer measurement. Based on SEM surface morphology, n comparison to growth durations of 2 and 4 hours, a 6-hour growth period at 85°C results in ZnO nanorods with a more pronounced hexagonal wurtzite structure. The XRD data reveals a prominent 002 peak, indicating c-axis orientation in the ZnO nanorods.**

Keywords—wearable, energy harvesting, piezoelectric, zinc oxide nanorods

I. INTRODUCTION

Several industries, including healthcare, transportation, industrial automation, and environmental monitoring, have the potential to be revolutionized by energy harvesting technologies. It provides a sustainable and cost-effective method for powering low-power electronics and enabling wireless communication in remote and harsh environments. There are still many challenge to overcome in this field, such as improving the efficiency, reliability, and scalability of energy harvesting devices, despite the significant progress made [1].

There are several types of energy harvesters commonly used to capture energy from various sources such as solar energy, RF (Radio Frequency) energy harvester, vibration energy harvester and bioenergy harvester. Solar energy harvesters, such as solar panels or photovoltaic cells, capture sunlight and convert it into electrical energy [2]. An RF energy harvester is capable of seizing and transforming electromagnetic waves or radio frequency signals into electrical power, enabling it to gather energy from the surrounding RF environment, such as Wi-Fi signals or mobile networks [3]. Additionally, bioenergy harvesters draw energy from biological origins, such as the human body or living organisms. Instances include wearable gadgets that collect energy from bodily motions and microorganisms that generate electrical power through metabolic processes [4].

Vibration energy harvesting provides a means to generate electricity from ambient vibrations that are often wasted or underutilized. By harnessing these vibrations, it enables the generation of electricity without depending on conventional energy sources such as fossil fuels, thus contributing to a reduction in our environmental impact and the advancement of sustainable energy methods [5]. One of the key advantages of vibration energy harvesting is the potential for creating self-powered systems. Many small electronic devices, wireless sensors, and IoT (Internet of Things) devices require a reliable power source [6]. This study focuses on piezoelectric energy harvesting because it can efficiently capture and convert the abundant mechanical energy from human motions such as walking and running into electricity. In addition, piezoelectric materials have an efficient energy conversion rate. When mechanical stress is applied to these materials, a voltage potential can be generated across their surface, making them ideal for harvesting energy from mechanical motions. This efficiency assures that even small-scale mechanical actions can generate significant amounts of energy.

Various piezoelectric materials have been utilized to generate piezoelectricity via mechanical vibrations. Due to its presence in various one-dimensional nanostructures, Zinc Oxide (ZnO) semiconducting material is regarded as a promising piezoelectric material and an essential step in the development of self-powered nanogenerator [7]. ZnO is an abundant natural metal oxide semiconductor with a wide bandgap, low cost, chemical stability in air, and biological safety. In addition, it is biocompatible, inherently piezoelectric, and has affordable synthesis and fabrication possibilities, making it ideal for energy harvesting applications.

II. Designs

A. Design Specification for Energy Harvester on Textile

Design requirements for a piezoelectric device depend on the specific application and intended functionality. The device should be mechanically robust to withstand the applied stress, strain, or deformation. For wearable energy harvester, the device should exhibit high sensitivity to the applied mechanical stimuli, enabling efficient energy conversion or accurate sensing capabilities. For this work, the device will be placed on the textile cloth as shown in figure in Figure 1 and the vibration generated by human motion will produce electricity that can be harvested.

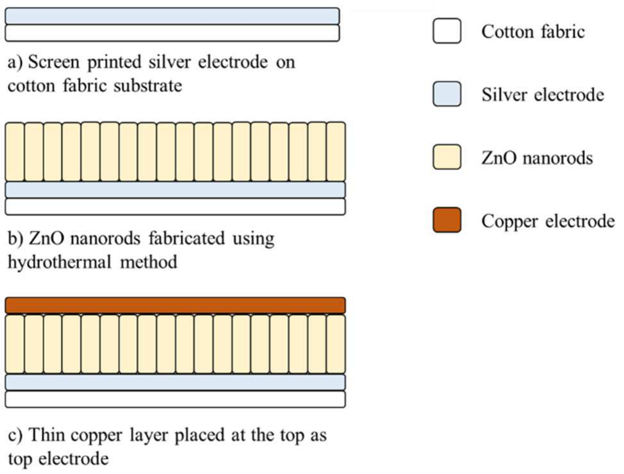


Fig. 1. Cross-section amd fabrication sequencs for ZnO energy harvester on textile.

Several design iterations were conducted to identify the optimal parameters for the piezoelectric energy harvester. The devices were specifically developed as wearable energy harvesters, intended to capture energy from walking and running motions. These activities are known to exhibit frequencies ranging from 2Hz to 12Hz, as reported by Sazonov et. al [8].

B. Equivalent Circuit Considerations

A lumped parameter electronic circuit model was used to simulate the interaction of piezoelectric devices with electronic circuits attached to them. Equivalent circuits for piezo generators and vibrational sensors operating quasi-statically are simplest form of useful model.

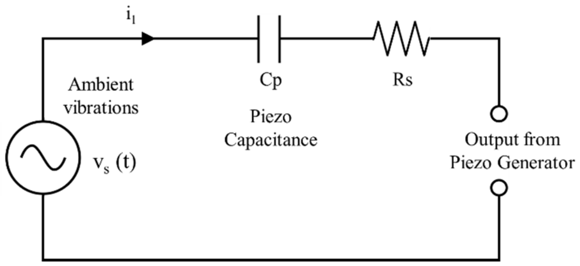


Fig. 2. Piezo transducer modeled as a voltage generator

Figure 2 shows a piezo device modeled as a voltage generator. Voltage output V is the open circuit voltage and proportional to the stress applied to the piezo device Cp is the lab measured capacitance of the piezo sensor. Rs, the equivalent series resistance of the sensor, is frequently taken as zero.

C. Theoretical Calculation for ZnO Nanorod

When a uniaxial compressive strain is exerted, the ZnO wurtzite cells experience deformation, resulting in the generation of an electric charge at the ends of the nanorod. Consequently, a piezo potential is formed along the c-axis of each cell, resembling a dipole. The relationship between polarization field produced by the stress can be described as follows:

$$D = d \times T \tag{1}$$

$$Q = C \times V \tag{2}$$

where D is the polarization, d is the piezoelectric stress coefficient and T is stress. For the electrostatic equation (ii), Q is the charge of the capacitor, C is capacitance value and V is the voltage. The capacitance equation determined by the permittivity of the material under free stress can obtained the final value of the electric potential by:

$$V = \frac{d \times F \times t}{\varepsilon_r \varepsilon_0 A} \tag{3}$$

where ε_r is relative permittivity, ε_0 is vacuum permittivity and t is the thickness of the piezoelectric material. Based on derived equation, the area of the piezoelectric material increases, the voltage will decrease.

III. Material and Methods

A. Textile Preparation – Fabrication of Silver Electrode

Screen printing is widely used printing technique that involves transferring ink through a mesh screen onto a substrate. Several preparations are necessary for a successful screen-printing process, including screen mesh design, ink and substrate selection, printing parameters, and curing techniques to ensure optimal adhesion of ink to the substrate.

A polyester mesh 230 thread per inch with 20 µm emulsion thickness and a 25 N tension screen mesh was used for screen printing of the electrode to the fabric using an automatic DEK printer at MTI Lab, Jabil Circuit Sdn. Bhd. Penang. Prior to printing, silver ink with 70% metal loading (AST6400) from Asahi Group was mixed and defoamed at 600 rpm and 700 rpm respectively for 30 seconds using a centrifugal mixer (Thinky Mixer Model ARE-310). This is to ensure the ink is homogenous and to get rid of trapped air. The silver ink was cured in a conventional oven (Contherm Thermotec 2000) at 120 °C for 45 minutes.

B. Zinc Oxide Nanorods Synthesis

Dip coating or hydrothermal method is widely used for coating fabrics and textiles as it offers a simplest way where involves immersing the substrate into a solution. Hydrothermal method was implemented to synthesize ZnO nanorods on the cotton fabrics. There are a few important things that need to be prepared for hydrothermal process include magnetic stirrer with heater, chemical reagents of ZnO nanorods, and binder oven for curing to ensure the adhesion of ZnO to the substrate. Synthesize ZnO nanorods on the cotton fabric involved 4 stages which is cleaning, seeding, growing, and annealing as shown in Figure 3.

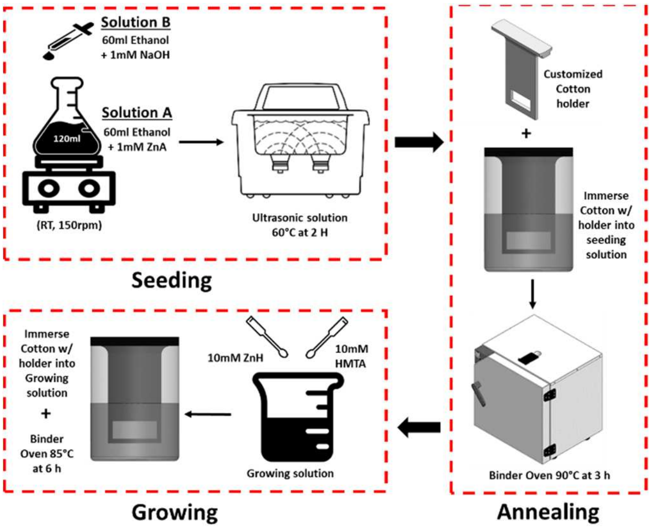


Fig. 3. Hydrothermal fabrication sequence process

To prepare a seeding solution, zinc acetate, sodium hydroxide, and ethanol were employed. Zinc acetate served as the precursor ion source and was dissolved in 60 mL of ethanol, stirred at 60°C on a magnetic hot plate to generate a Zn precursor. Separately, sodium hydroxide (1 mM) was dissolved in 60mL of ethanol, stirred for 15 minutes, and then cooled. Once both solutions had reached room temperature, 60 mL of ethanol was combined with the Zn precursor through stirring. To potentially impact the morphology of ZnO nanostructures, 2 mL of sodium ethoxide was gradually added to the Zn precursor in the form of droplets while maintaining the pH level between 8 and 9. The resulting ZnO precursor underwent ultrasonication at 60°C for a duration of two hours using Crest Ultrasonics, resulting to a cloudy solution indicative of the presence of the ZnO precursor.

3 samples of ZnO nanorods on top of silver electrode was fabricated with varies the temperature and growing time to observe the different structure of nanorods. Sample A, B and C have a growing time and temperature at 6-hours 85 °C, 4-hours 140 °C and 2-hours 180 °C respectively.

The cotton textile with the customized cotton holder was immersed in the ZnO precursor vertically at 90°C for 3 hours as to produce a uniform ZnO nanorod arrays. The annealing process of the sample can be placed on hot plate or in the oven. In the final stage of the low-temperature hydrothermal method, ZnO-nanorod particles were grown within the seeded region of the cotton. The growth solution was prepared by mixing 10 mM of zinc nitrate hexahydrate, 10 mM of hexamethylenetetramine, and 1000 mL of deionized water and stirring until homogeneous. Then the cotton was immersed in the growth solution and placed in an oven kept at 85°C for 6 hours for Sample A. For Sample B, the growing time was changed to 4 hours at 140°C and then Sample C was prepared with 2 hours growing time at 180°C. After the growth period, the cotton was removed from the solution and washed with deionized water to remove excess impurities.

C. Characterization of ZnO nanorod

Multiple samples of ZnO nanorod on textile were prepared to observe the surface morphology and the characteristic of piezoelectric material. For the surface morphology of fabric, the observation was performed by scanning electron microscopy, SEM (JEOL JSM-IT100) at the magnification 4000×. The XRD was applied to study the crystalline structure of ZnO nanorods, using PANalytical XPert Pro 3. Cu-Kα radiation was used, the scanning speed was set to 0.05°C/s, and a 2θ scan range was fixed from 20° to 60°.

IV. Result and Discussion

From the 3 samples that was fabricated with varies the growing time and temperature, Sample A shows the successful fabrication of ZnO nanorods compared to Sample B and C. The cotton fabric that has been fabricated was cut into small size 1cm x 1cm before proceeding with the characterization. The resistance value of Sample A was measured at the silver electrode (R = 0.4Ω) and across the growth of ZnO nanorods (R = 1.1Ω) as shown in Figure 4.

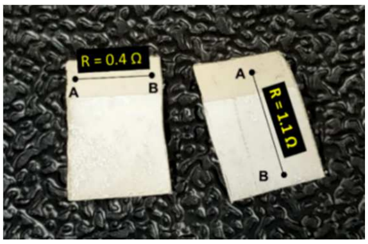


Fig. 4. The ZnO piezoelectric device was cut and measured the resistance value.

A. SEM characterization

The morphological properties of the ZnO nanorod on cotton fabric were studied by scanning electron microscopy (SEM). SEM was used to visualize and analyze the ZnO nanorod layer growth on the textile substrate. SEM provides high-resolution images with a large depth of field, allowing to examine the morphology, structure, and composition of materials at a nanoscale level.

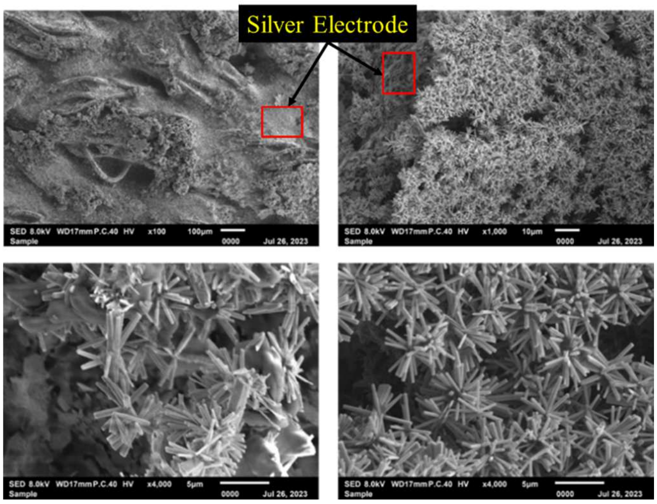
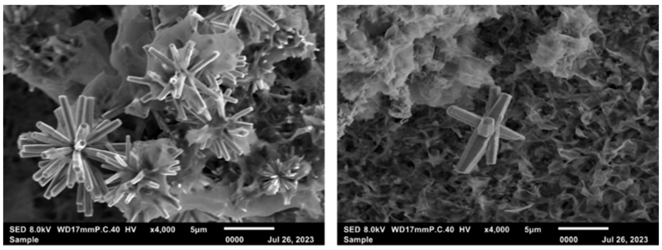


Fig. 5. Results of SEM characterization for Sample A at different magnification.

Figure 5 shows the SEM characterization morphology for Sample A with different magnification images varies at x100, x1000 and x4000. At magnification x100, a clear image of textile thread with ZnO nanorod attached on top of silver electrode. Surface morphology at x4000 magnifications, it can be observed that the ZnO nanorods were fully grown on top of the silver electrode with the length of the nanorods approximately around 4 μm.



(a) (b)

Fig. 6. Results of SEM characterization at magnification of x4000 for (a) Sample B (b) Sample C

Based on SEM morphology for Sample B and Sample C shown in Figure 6, it was observed that 2-hours growth period resulted in a few ZnO nanorods and together with the ZnO nanoparticles that are underdeveloped not fully grown. For 4-hours growth period, the ZnO are fully grown but like gel structure appear around the ZnO nanorods. Through the hydrothermal process, a solution or gel containing zinc ions and other reactants is used. As the reaction proceeds with low time of growth duration, ZnO nanorods grow within the gel matrix are not fully developed.

B. XRD characterization

The crystal structure properties of the ZnO nanorods have been characterize with the XRD spectroscopy pattern at room temperature in the range of 2θ as shown in Figure 7. Fig 7 (a) shows the XRD pattern with only cotton fabric and silver electrode without ZnO nanorods, meanwhile Fig 7 (b) shows the XRD pattern with the presence of ZnO nanorods structure.

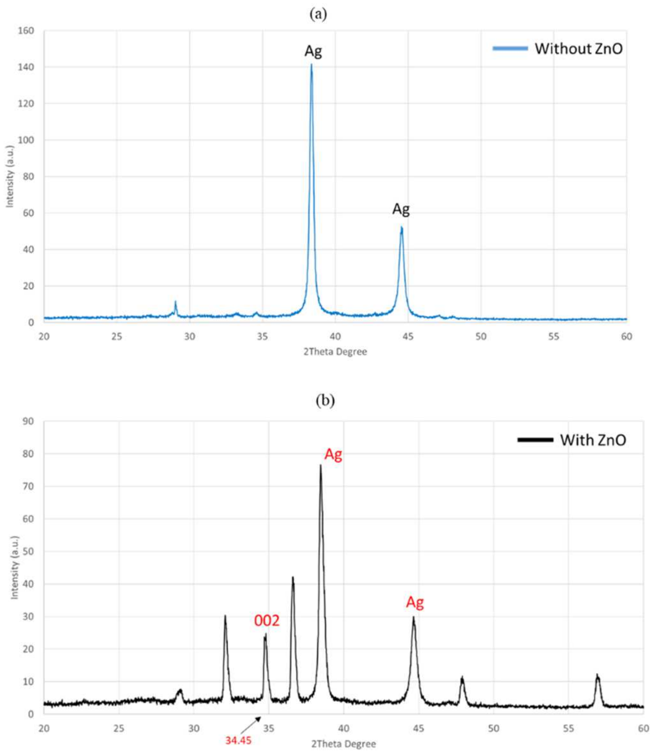


Fig. 7. (a) XRD pattern without ZnO nanorods (b) XRD pattern with ZnO nanorods.

The XRD results shown that the ZnO nanorods have a hexagonal wurtzite structure (JCPDS card no: 01-082-9744) at the peak of 34.45° that grow along the (002) which indicate ZnO nanorods are well c-axis oriented. The peaks at 2θ value of 38.45° and 44.76° are the diffractive peaks of silver electrode on cotton fabric.

V. Conclusion

In conclusion, the synthesis of ZnO nanorods on the silver electrode is successful. Fabrication of the ZnO has been discussed. A screen-printing method was used to coat the surface of cotton fabric with silver ink to act as an electrode of piezoelectric device. The morphology of the ZnO has been characterized by SEM and crystal structure analysis was done by XRD measurement. This research has highlighted the piezoelectric design and equivalent circuit with the theoretical equation. By controlling the growing time and the temperature of the growth precursor solution, the ZnO nanorods arrays can be differ in term of the structure. The SEM results indicate that the growing time of 6 hours at 85 °C have a better hexagonal wurtzite structure of ZnO nanorods compared to 2- and 4-hours growing time. The XRD results shows the 002 peak where the ZnO nanorods are well c-axis oriented. Future work with these results will be focused on electromechanical measurement of the piezo device and another characterization such as EDS and piezometer d_{33} to validate the performance of piezoelectric for wearable devices.

Acknowledgment

This project is sponsored by the Asian Office of Aerospace Research and Development (AOARD) under grant project SPI21-082-0082. The authors would like to thank Jabil Circuit Sdn. Bhd. for funding the research materials and equipment.

References

[1] S. Priya and D. J. Inman, *Energy harvesting technologies*, vol. 21. Springer, 2009.

[2] Z. Abdin *et al.*, "Solar energy harvesting with the application of nanotechnology," *Renewable and sustainable energy reviews*, vol. 26, pp. 837–852, 2013.

[3] S. K. Divakaran, D. Das Krishna, and Nasimuddin, "RF energy harvesting systems: An overview and design issues," *International Journal of RF and Microwave Computer-Aided Engineering*, vol. 29, no. 1, p. e21633, 2019.

[4] I. Jeerapan, J. R. Sempionatto, and J. Wang, "On-body bioelectronics: wearable biofuel cells for bioenergy harvesting and self-powered biosensing," *Adv Funct Mater*, vol. 30, no. 29, p. 1906243, 2020.

[5] C. Wei and X. Jing, "A comprehensive review on vibration energy harvesting: Modelling and realization," *Renewable and Sustainable Energy Reviews*, vol. 74, pp. 1–18, 2017.

[6] S. Fang, S. Wang, S. Zhou, Z. Yang, and W.-H. Liao, "Exploiting the advantages of the centrifugal softening effect in rotational impact energy harvesting," *Appl Phys Lett*, vol. 116, no. 6, p. 063903, 2020.

[7] Zhao, M. H., Wang, Z. L., & Mao, S. X. (2004). Piezoelectric characterization of individual zinc oxide nanobelt probed by piezoresponse force microscope. Nano Letters, 4(4), 587-590.

[8] N. Hegde, M. Bries, and E. Sazonov, "A comparative review of footwear-based wearable systems," *Electronics (Basel)*, vol. 5, no. 3, p. 48, 2016.