

Simple Fabrication and Characterization of Piezoelectric Nanogenerators from Cobalt-Doped Zinc Oxide Nanofibers

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Abstract: The ferromagnetic cobalt doped into Zinc Oxide (ZnO) crystals is a promising method for enhancing the performance of Piezoelectric-Based Nano Generators (PENGs). The study reports the characterization and testing PENGs which simply fabricated from cobalt-doped ZnO nanofibers (NFs) synthesized by electro spinning machine. The electro spinning processes were conducted at a flow rate of 4 $\mu\text{L}/\text{min}$ and at various cobalt concentrations followed by sintering at a temperature of 500°C. The X-Ray Diffraction (XRD) spectra demonstrate that the crystalline diameter of Co-doped ZnO NFs increased as the content of cobalt increased, up to a concentration of 9%. Meanwhile, the maximum output voltage and power density of PENGs with Co-doped ZnO NFs were 221.6 mV and 148.8 nW/cm^2 , respectively when the PENGs were subjected to a cyclic mechanical load of 0.5 kg. Therefore, PENGs fabricated from Co-doped ZnO NFs are challenging for the next generation of self-powered devices.

Keywords: Fibers, Cobalt, Doping, Zinc Oxide, Electro Spinning, Piezoelectric, Nanogenerator

Introduction

Piezoelectric Nanogenerators (PENGs) have been investigated by many studies because of their capability to harvest mechanical energy from human motions, finger taps and environment movements into electrical energy via piezoelectric materials. PENGs have also been employed in various devices including wireless sensors, self-powered equipments and electromechanical systems (Cho *et al.*, 2017). The key issues in PENGs are the design and the materials used (Dagdeviren *et al.*, 2016).

There are various materials having a non-centrosymmetric property exploited for PENGs. Among them, ZnO materials are very promising because of its abundant availability, environmental friendliness and high Curie temperature. Above the Curie temperature, a non-centrosymmetric crystal transforms into a centrosymmetric structure and both ferroelectric city and piezoelectricity disappear (Ubaidillah *et al.*, 2013; Xing *et al.*, 2015).

The piezoelectricity of materials was represented by the piezoelectric constant, d_{33} . The higher the

piezoelectricity, the bigger the voltage is generated from the PENGs. ZnO Nanowires (NWs) have a slightly bigger piezoelectric constant d_{33} of 15 pm/V (Christian *et al.*, 2016) than that of the bulk ZnO with d_{33} of 9.93 pm/V (Zhao *et al.*, 2004). ZnO NWs have a piezoelectric constant ranging from 1 to 75 pm/V (Lee *et al.*, 2012) depending on their morphology. Meanwhile, ZnO Nanobelts (NBs) have bigger d_{33} (14.3-26.7 pm/V) than that of ZnO Thin Films (TFs) synthesized by sputtering deposition (14 pm/V) (Dagdeviren *et al.*, 2013). However, the ZnO TFs deposited onto a Si substrate by pulsed laser ablation has a high piezoelectric constant of 49.7 pm/V (Qin *et al.*, 2016a; 2016b) because it exhibited a preferred c-axis orientation. In addition, ZnO Nanorods (NRs) were reported have the d_{33} ranging from 4.41 to 11.8 pm/V (Tamvakos *et al.*, 2015) for single crystal and they further reached a highest value of 26.7 pm/V when measured for ZnO NBs (Zhao *et al.*, 2004). Therefore, the morphology of ZnO significantly affects to the piezoelectric property, d_{33} .

It has also been revealed that doping has an important effect on both a structural and a piezoelectric constant of

ZnO. Piezoelectric constant evaluated for ZnO TFs doped with Ni, Cu, Fe and Co shows a variation in the d_{33} values ranging from 5 to 14 pm/V (Zamiri *et al.*, 2016). A significant increase in d_{33} values up to 110 pm/V has been reported for V-doped ZnO TFs (Zamiri *et al.*, 2016). Meanwhile, Cr-doped ZnO NRs have a very small d_{33} value of 1.68 pm/V (Sinha *et al.*, 2014). Besides, Europium-doped ZnO NRs have the mean d_{33} of 43.38 pm/V (Yadav *et al.*, 2016). Therefore, the morphology and doping play important role for enhancing the performance of PENGs based on ZnO nanomaterials. However, to the best of our knowledge, there are only few studies on the piezoelectric constant of cobalt-doped ZnO Nano Fibers (NFs) and used for PENGs.

PENGs with a hetero-junction material of Li-doped $\text{Cu}_2\text{O}/\text{ZnO}$ produced a power of 52 μW (Cho *et al.*, 2017). The Li-doped $\text{Cu}_2\text{O}/\text{ZnO}$ synthesized by radio frequency magnetron sputtering was flexible and transparent. Moreover, PENGs with the ZnO TFs deposited onto a Si substrate by pulsed laser ablation at a substrate temperature of 500°C generated a voltage, current and power of 95 mV, 35 $\mu\text{A}/\text{cm}^2$ and 5.1 mW/cm^2 , respectively (Qin *et al.*, 2016a; 2016b). The PENGs with Ga-doped ZnO NWs produced a higher voltage ranging from 120 to 560 mV (Zhao *et al.*, 2015b) if compared with undoped ZnO NWs generated a lower voltage of up to 25 mV (Wang *et al.*, 2008). Meanwhile, PENGs fabricated from Al-doped ZnO NRs and NFs capable of generating a voltage up to 60 mV (Fang and Kang, 2010) and 265.5 mV (Suyitno *et al.*, 2014b), respectively. However, the voltage and power density of PENGs with ZnO nanomaterials are still low (Ren *et al.*, 2009; Kanjwal *et al.*, 2011).

Among the aforementioned materials used for PENGs, the ZnO NFs are very interesting because NFs can be simply synthesized using a low cost electrospinning machine (He *et al.*, 2008) with a good durability (Suyitno *et al.*, 2014a). Unfortunately, ZnO NFs exploited for PENGs are still limited. Besides Al, Ga, Li, Ni, Cu, Fe and Cr doping; Co-doped ZnO NFs synthesized by electrospinning method is a promising for enhancing the properties of ZnO-based piezoelectric materials fabricated for PENGs. There is a study reported that cobalt doping does not significantly affect their piezoelectric performance, although it induces the paramagnetic state and gives rise to new photoluminescence emission peaks because of small changes in the lattice parameters and no changes to crystal structures (Zhao *et al.*, 2015a). However, cobalt doping into ZnO nanoparticles (NP) synthesized by a co-precipitation method decreased a crystallite size and changed the diamagnetic behavior of ZnO into ferromagnetic nature (Gandhi *et al.*, 2014).

Meanwhile, another study reported that the lattice constants of the c-axis increased with increasing cobalt content up to 25% and decreased above 25% because of the solubility of cobalt exceeds a thermal equilibrium limit below 25% due to a no-equilibrium sol-gel process (Lee *et al.*, 2002).

The aforementioned studies show that the effect of cobalt doping into ZnO materials is still controversy and need to be deeply investigated particularly for ZnO NFs synthesized by electrospinning method. Furthermore, the optimal concentration of Co doping into ZnO NFs applied for PENGs has not been entirely studied and characterized. Therefore, we report here the simple fabrication and the characterization of PENGs from cobalt-doped zinc oxide fibres synthesized by electrospinning method. In addition, we evaluate also the influence of the cobalt content on the performance of PENGs simply fabricated from cobalt-doped ZnO NFs.

Method

Fabrication PENGs

PENGs were simply fabricated from Co-doped ZnO NFs, two electrodes and Polydimethylsiloxane (PDMS) polymer. The NFs were synthesized by electro spinning method (Suyitno *et al.*, 2014b). To avoid the damage of NFs, the collector used in electro spinning method was aluminum plate and then subsequently used for electrode. Both electrodes were glued together with PDMS as show in Fig. 1.

Synthesizing Co-Doped ZnO NFs

The materials used for synthesizing the Co-doped ZnO NFs were polyvinyl alcohol ($[-\text{CH}_2\text{CHOH}-]_n$, PVA, 72000 MW, Merck), zinc acetate dihydrate ($\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$, ZnAc, Merck) and cobalt(II) acetate tetrahydrate ($(\text{CH}_3\text{COO})_2\text{Co} \cdot 4\text{H}_2\text{O}$, CoAc, Merck) and used as received.

We first synthesized the PVA solution. The PVA was dissolved in purified water at a mass ratio of 1:10, stirred at a temperature of 70°C for 4 h and allowed to settle for 24 h at a temperature of 25°C.

To synthesize the CoAc/ZnAc solution with a cobalt content of 8%, 0.32 g of CoAc, 4.0 g of ZnAc and 16 g of H_2O were mixed and stirred by magnetic stirrer at a temperature of 70°C for 2 h. Next, synthesizing the CoAc/ZnAc/PVA solution was conducted by mixing 1 g of the CoAc/ZnAc solution with 5 g of PVA solution, stirred at a temperature of 70°C for 2 h and followed by settling for 24 h at a temperature of 25°C. The aforementioned procedures were repeated for synthesizing the CoAc/ZnAc/PVA solution at a cobalt content of 4, 6, 8, 10, 12 and 14%.

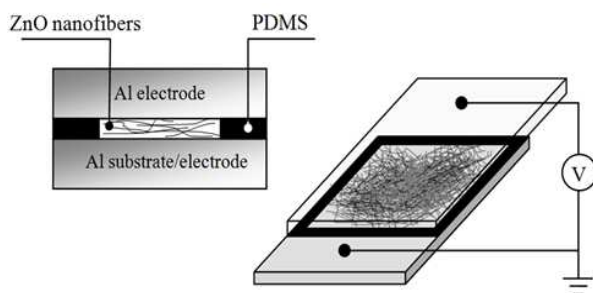


Fig. 1. PENGs configuration

In addition, 0.5 mL^{-1} of CoAc/ZnAc/PVA solution was poured in the syringe pump and electrospun in a horizontal electro spinning machine to produce the green fibers consisting of cobalt-doped ZnAc/PVA. The needle diameter of the syringe pump is 0.6 mm. The distance between the tip needle of the syringe pump and the collector of an aluminum plate is 8 cm. When a high voltage of approximately 15 kV was applied, the solution was pulled out by electrostatic fields, attached on the surface of an aluminum collector plate and finally produced the non-woven green fibers. The green fibers were then sintered for 4 h at a temperature of 500°C (Yang *et al.*, 2004) to remove PVA or organic materials and to growth the crystal of Co-doped ZnO fibers.

Characterizing ZnO NFs and Testing PENGs

The crystalline and morphological structures of Co-doped ZnO NFs were characterized using X-Ray Diffraction (XRD) and scanning electron microscopy (SEM, FEI: Inspect-S50). The analysis of SEM images was conducted by image software from Image J.

The piezoelectric constant d_{33} of Co-doped ZnO NFs was measured by d_{33} Piezometer Systems from Piezotest Pte Ltd. Meanwhile, the PENGs were measured their output electric energy using an ADAM Advantech (series 4018) and software PCLS-Adam View 32. When measuring the output electric energy from NGs, the cyclic mechanical load of 0.5 kg was applied to represent the condition of a light finger tapping (Choi *et al.*, 2010). The power of piezoelectric-based NGs was subsequently calculated from the output voltage generated by the variation of load resistance (Ω) (Chen *et al.*, 2010), as following:

$$P_L = \frac{1}{T} \int \frac{V_o(t)^2}{R_L} dt \quad (1)$$

Where:

- P_L = The output power (W)
- T = The period (s)
- V_o = Is the output voltage (V)
- t = time (s)
- R_L = Is the electric load (Ω)

Results and Discussion

Structural Analysis

Figure 2 shows the spectra of X-ray diffraction of the fibers of Co-doped ZnO NFs with cobalt content from 0 to 14%. The sharp diffraction peaks show the good crystalline quality. The NFs tested showed similar diffraction patterns with the highest peak between 35.88 and 36.15° , which is the typical peak of zincite ZnO crystal (101). The diffraction patterns of Co-doped ZnO NFs corresponded to the data from JC-PDS standard card (PDF#36-1451, space group P63 mc (186)).

Meanwhile, the shifts in the angle of diffraction peaks indicated the impurities of ZnO crystals caused by cobalt doping. Moreover, Fig. 2 shows also that clear sharp peaks coinciding with other impurities did not appear, suggesting that new phases did not arise and cobalt atoms were substituted within ZnO crystal lattices (Feng *et al.*, 2009). At higher cobalt content, the peaks of diffraction appeared sharper which is in agreement with previous study (Nirmala and Anukaliani, 2011). In contrast, at a cobalt content of 14%, the diffraction peaks were smaller and no peaks of cobalt compounds or cobalt existed, indicating the crystallization process was inhibited by means of impurity atoms.

Meanwhile, the crystalline diameter of cobalt-doped ZnO fibers was calculated using the Debye-Scherrer equation (Pongwan *et al.*, 2012; Asikuzun *et al.*, 2015):

$$D = \frac{k\lambda}{B\cos\theta} \quad (2)$$

Where:

- D = The crystalline diameter
- λ = The wavelength of X-ray ($\text{CuK}\alpha = 0.15406 \text{ nm}$)
- k = The constant (0.9)
- θ = The diffraction angle
- B = The full width at half maximum.

Table 1 shows that the crystalline diameter of Co-doped ZnO NFs were influenced by the concentration of cobalt. The corresponding crystalline diameter of Co-doped ZnO NFs increased by almost twice at a cobalt concentration of 9%, which declined as the cobalt content further increased. The increase of grain size at a certain concentration of doping cobalt was also revealed from previous studies (Lee *et al.* 2002; Bahadur *et al.*, 2010; Benramache *et al.*, 2013; Gandhi *et al.*, 2014; Asikuzun *et al.*, 2015). Therefore, the previous study from (Zhao *et al.*, 2015a) stating that cobalt doping does not significantly affect the lattice parameters and no changes to crystal structures is misleading within the synthesis of Co-doped ZnO NFs by the electrospinning machine.

Furthermore, the crystalline diameter of Co-doped ZnO NFs varied ranging from 9.0 to 17.7 nm. Doping cobalt at a concentration higher than 9% affected the decrease of the crystalline diameter because more cores were formed during the crystallization process (Lee *et al.*, 2008).

Meanwhile, Fig. 3 shows the SEM images of the Co-doped ZnO NFs at a magnification of 50,000 \times . It can be observed that the fibers are randomly arranged. As shown in Table 2, the diameter of the fibres increased as the cobalt content increased up to a concentration of 9%. The average diameter of the Co-doped ZnO NFs ranged from 87 to 133 nm.

Performance of PENGs from Co-Doped ZnO NFs

The piezoelectric constant of Co-doped ZnO NFs has been measured and shown in Table 3. There is a slight

increase of d_{33} as increasing concentration of doping Co up to 9-10%. The d_{33} values of Co-doped ZnO NFs ranging from 3.7 to 4.1 pm/V which is comparable with that of ZnO NRs ranging from 4.41 to 11.8 pm/V (Tamvakos *et al.*, 2015) and a bit higher than that of Cr-doped ZnO NRs (1.68 pm/V) (Sinha *et al.*, 2014).

Meanwhile, testing performance of NGs was conducted by applying the cyclic mechanical load of 0.5 kg every 3 sec. When the c-axis of cobalt-doped ZnO fibers was under the applied deformation, a piezoelectric field was created along fibers, which drive the electrons flowing in the external circuit.

Figure 4 shows the output voltage (mV) produced from PENGs. Meanwhile, Fig. 5 shows the output power (nW) of PENGs. The maximum power and the maximum voltage produced by the PENGs can be seen in Table 4.

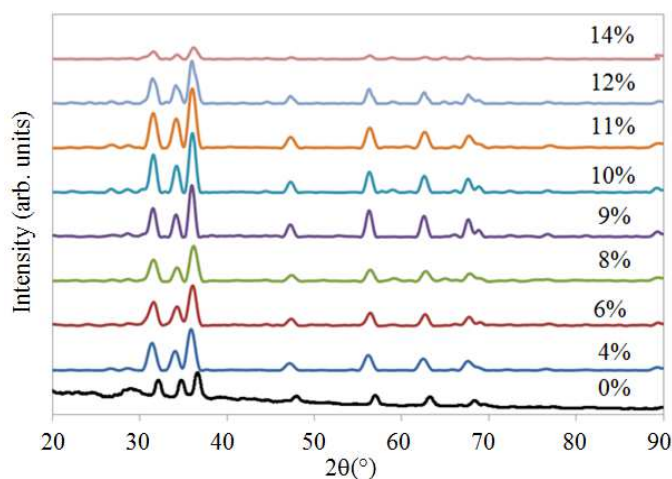


Fig. 2. X-ray diffraction patterns of cobalt-doped ZnO fibers

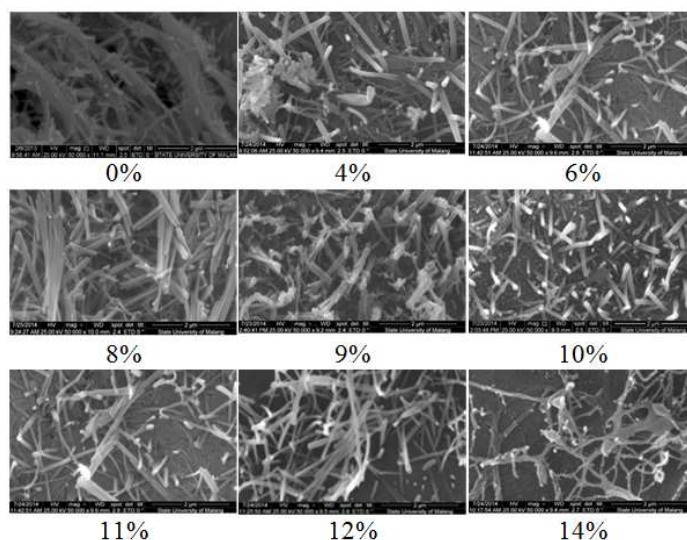


Fig. 3. SEM images of cobalt-doped ZnO fibers

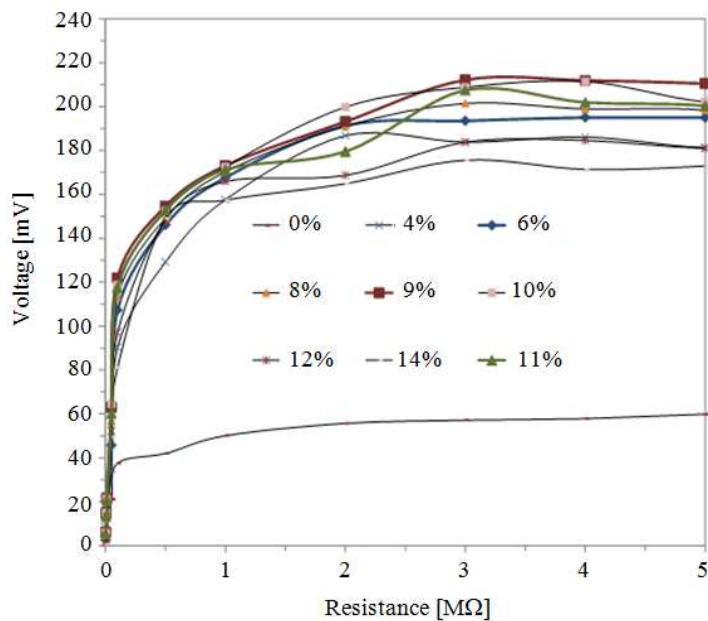


Fig. 4. Output voltage of PENGs from Co-doped ZnO NFs at various concentrations of cobalt

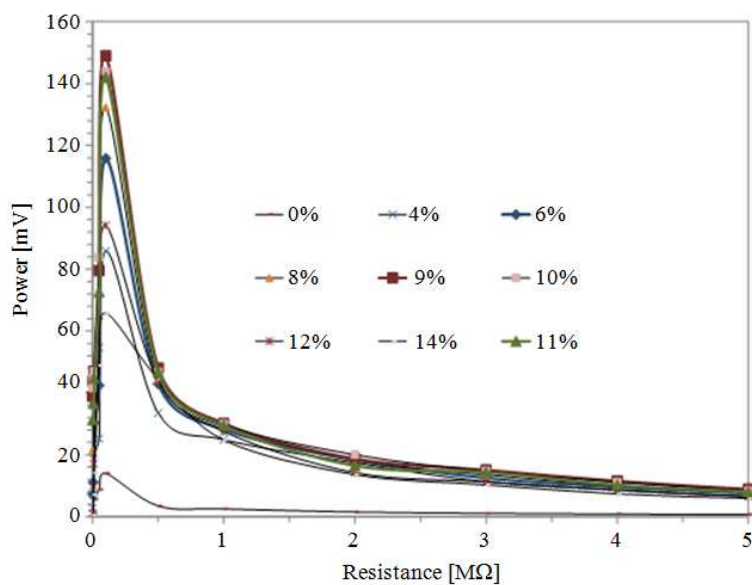


Fig. 5. Output power of PENGs from Co-doped ZnO NFs at various concentration of cobalt

Table 1. Crystalline diameter and crystallinity of cobalt-doped ZnO fibers

Cobalt concentration	2 theta	FWHM	Crystalline diameter (nm)
0%	35.886	0.933	9.0
4%	35.880	0.888	9.4
6%	36.148	0.851	9.8
8%	36.119	0.746	11.2
9%	35.934	0.471	17.7
10%	36.060	0.603	13.9
11%	36.013	0.633	13.2
12%	36.061	0.742	11.3
14%	36.151	0.808	10.3

Table 2. Diameter of cobalt-doped ZnO fibers

Fiber diameter (nm)	Doping concentration (%)								
	0	4	6	8	9	10	11	12	14
Maximum	283	169	204	208	304	202	199	164	250
Minimum	42	39	37	40	33	50	47	26	37
Mean	91	93	96	110	133	102	88	87	87
Standard deviation	50	40	39	42	77	35	32	31	52

Table 3. Piezoelectric constant, d_{33} , of Co-doped ZnO NFs

Cobalt concentration	d_{33} (pm/V)
0%	3.7
4%	3.7
6%	3.8
8%	3.9
9%	4.1
10%	4.1
11%	4.0
12%	4.0
14%	3.9

Table 4. Maximum power and voltage produced by the piezoelectric-based NGs

Performance	Cobalt concentration									
	0%	2%	4%	6%	8%	9%	10%	11%	12%	14%
Max voltage [mV]	64.2	180.2	189.9	205.3	214.1	221.6	220.6	218.9	201.9	198.1
Max power [nW]	14.0	42.9	85.9	115.8	132.4	148.9	144.0	142.0	94.2	65.7

Piezoelectric material is materials applied loads produces electricity. At a cyclic mechanical load of 0.5 kg, the output voltage generated by PENGs with Co-doped ZnO NFs was influenced by the resistance loads. The output voltage generated from PENGs increased as the concentration of cobalt doping increased. The sharp increase of output voltage was generated when the resistance load increased up to 1 k Ω , followed with slight increase at higher resistance load than 1 k Ω . At doping cobalt about 9%, the output voltage generated increased nearly 3.45 times than that produced by PENGs without doping cobalt.

The maximum output voltage of PENGs was 221.6 mV. The output voltage of the PENGs with Co-doped ZnO NFs was higher when compared with previous study (Choi *et al.*, 2010). PENGs with ZnO TFs generated voltages of 20 and 60 mV at a mechanical load of 0.4 and 1 kg, respectively. In contrast, the PENGs based on NiO/ZnO NWs were capable to generate a voltage of 388 mV (Qu *et al.*, 2016) because ZnO NWs were vertically grown. Meanwhile, by ferrous doping, PENGs with the vertical ZnO NWs generated a voltage up to 850 mV (Zhu *et al.*, 2015).

In addition, the output power generated by PENGs with Co-doped ZnO NFs increased as the cobalt concentration increased up to 9%. As previously discussed, the synthesized NFs demonstrated the highest crystallinity at a cobalt concentration at 9%. The maximum output power of PENGs, 148.9 nW, was generated when the cobalt concentration of 9% as shown

in Fig. 5 corresponding to an output current of 1.2 μ A. The current generated from the PENGs with Co-doped ZnO NFs was greater than that from the PENGs with ZnO NWs/PZT (lead zirconate titanate) hetero-junction, namely 270 nA (Im-Jun *et al.*, 2013) and with NiO-ZnO hetero-junction, namely 40 nA/cm² (Yin *et al.*, 2015). Moreover, the maximum output power was equivalent to a power density of 148.8 nW/cm². The power density produced by PENGs with Co-doped ZnO NFs was greater than the achieved by a previous study, namely 51.7 nW/cm² (Suyitno *et al.*, 2014b).

Conclusion

PENGs were simply fabricated from Co-doped ZnO NFs and successfully tested. The greatest crystallinity was achieved by cobalt doping at a concentration of 9%. Furthermore, the cobalt concentration has doubled the crystalline diameter of ZnO NFs. In contrast, the cobalt content in the ZnO NFs significantly influenced the output voltage and output power of PENGs. The maximum output voltage of PENGs with Co-doped ZnO NFs is 221.6 mV. Meanwhile, the maximum output power, which is equivalent to the power density of 148.8 nW/cm², was achieved when PENGs were subjected to a cyclic mechanical load of 0.5 kg. The PENGs fabricated from Co-doped ZnO NFs generated high power density values and are interesting candidates for employing in self-power devices.

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Author's Contributions

Suyitno: Principal investigator, reviewed the significant content and corresponding author.

Syamsul Hadi: Supervised the experiments, provided the exhaustive data and interpreted the data.

Mohd Zainizan Sahdan: Reviewed the related previous works, XRD analysis and checked the grammatical errors.

Nafarizal Nayan: Reviewed the logical writing, SEM analysis and checked the grammatical errors.

Hashim Saim: Reviewed the comprehensively discussion.

Agung Tri Wijayanta: Supervised the experiments, checked tables and figures and interpreted the data.

Lukman Nulhakim: Drafted the manuscripts and conducted the experiments of the performance of pems.

Ethics

The present work is not published in its present form in any journal or will not be published in any journal.

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