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Deposition of Multilayer Films of ZnO by Sol-gel Process on Stainless Steel Substrates for Energy Harvesting Devices

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Abstract. The mechanical vibrations surrounding the environment can be converted in electrical energy by piezoelectric energy harvesters (PEH). The increase on the availability of Wireless Sensor Networks (WSN) increases the need for power supply that replaces ordinary batteries. In this paper, an electromechanical modelling and a deposition of multilayer zinc oxide (ZnO) films for an PEH are presented. The aim of the study is to obtain the thickness of the multilayer ZnO films in different conditions, in order to improve the energy harvesting capacity for a PEH device. The ZnO synthesis was performed by the sol-gel method with dehydrated zinc acetate as a precursor, making deposits of 5, 10 and 15 layers. The depositions were made at room temperature by spin coating at 1440 revolutions per minute for 16 s. An UV-Vis test showed that ZnO were present, with a percentage of peak reflectance at 370 nm and a XRD test showed a preferential crystalline orientation at (002). Also, a Finite Element Modelling (FEM) simulation of the substrate behaviour was performed, functioning as a cantilever beam. When adding a seismic mass and oscillating in the resonance frequency of 103.31 Hz and with 15 layers of ZnO deposited, 2.67 volts were obtained.

1. Introduction

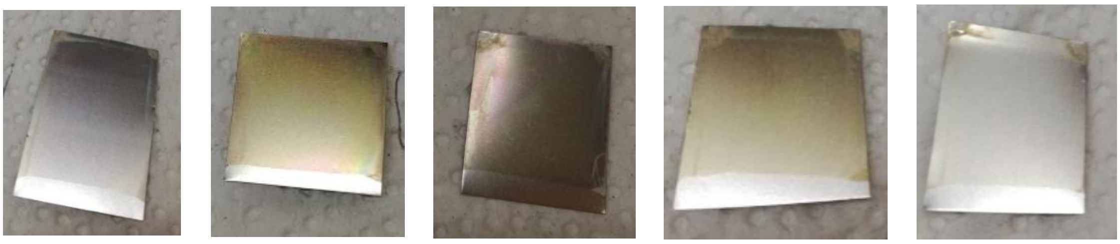
Nowadays, the power generation by fossil resources is the main cause of the climate change, showing an ecological footprint on the flora and fauna [1]. The current energetic demands appear to keep increasing, among various causes, by the rise of portable electronic devices. An Irish research group has reported that in the near future there will be 25 billion electronic devices constantly connected [2]. This increase is boosted by the concept of the Internet of Things (IoT), a trend in the current technology development. The IoT is expected to provide information from the surrounding environment in real time, using different elements interconnected with each other [2]. The micro and nanoelectromechanical systems (MEMS/NEMS) provides technology for the IoT network, like wireless sensors networks (WSN), actuators, data transfer devices, as well as the possibility of a ubiquitous installation and small size [3–5]. The IoT systems are used in different areas, which increase the need of mobile, wireless energy sources, commonly batteries are being used as energy storage device. Existing batteries have been studied for years improving their performance, nevertheless, they present deficiencies. For example, the dispositive portability has increasingly prioritized the design of electronic devices with the smallest dimensions and the least possible weight [6], both affected by the characteristics of the current batteries. Also, in some applications such as sensors for structural monitoring in remote locations, battery charging, or replacement could be costly or infeasible. Furthermore, an improper confinement of discarded batteries is a risk due to chemical substances such as mercury, cadmium, lithium or lead with which they are manufactured. For example, button cells' materials could be toxic to the flora and fauna of the planet, polluting the water. Advances in MEMS technology have allowed the production of small electric power generators as an option for energy supply. The energy produced by human activity like industrial production, the use of automobiles, bridges and buildings, among others, could be used as an energy source to generate electric power [7]. This energy sources are such as: mechanical vibrations, solar power, electromagnetic fields or thermal gradients. Among these energy options, sensitive mechanical vibrations are simple to use and can be exploited by energy harvesters (EH) [8]. The mechanical energy exists in most surroundings as consequence of human activities or natural resources, like breeze of the wind [9]. In addition to the ubiquity of this energy, it is possibility of design and manufacturing EH in the order of micrometers [10], which makes possibly to apply them with biocompatible portable sensors or energize sensors in bridges and buildings [8, 11].

Among the different type of EH, the Piezoelectric Energy Harvesters (PEH) generates relatively high-power density and voltage, which depends on the piezoelectric parameters of each different types of piezoelectric materials available. The principal section about the piezoelectric energy harvesters (PEH) is the piezoelectric material, a type of solid able to gather electric charge as result of applied mechanical stress, which is known as the piezoelectric effect [12]. In the PEH, the kinect energy could be provided from the mechanical vibration over the environment, which causes the desired stress on a determined object and generates electrical power. The zinc oxide (ZnO) is a material with piezoelectric properties that has been the subject of various investigations in recent years due to its physical properties [13]. It is a versatile material and can be deposited on a substrate using different techniques, for example the sol-gel method [14]. The application of different designs to supply energy to WSN are relevant duo the advantage of providing a mobile source of clean energy, with minimal maintenance and exploit the ubiquitous mechanical energy [15]. Hence, in this work, we present the procedure to obtain a multilayer ZnO film with a sol-gel synthesis method by variating the number of layers. Furthermore, a finite element modelling (FEM) simulation was performed to obtain an approximation of the energy that the obtained film could generate. A seismic mass was added to increase the obtained power [16] and decreased the resonant frequency of the PEH.

2. Methodology

The ZnO films were deposited by the methodology proposed by [17]. To synthesize ZnO solution, a Zinc acetate ehydrate was used as precursor, monoethanolamine (MEA) as stabilizer and 2-metoxyethanol as solvent. 14 ml of 2- methoxyethanol were put into a ball flask, and 430 µl of MEA were added and then this mixture was stirred for several minutes at room temperature. After that, 1.55 g of zinc acetate dihydrate was added to the previous solution, and stirred at room temperature for 30 min. The ZnO films was deposited on ANSI 302 stainless steel (SS302) substrates using the spin coating technique at 1440 revolution per minute (rpm) for 16 s. After each coating, the films were preheated in an oven at 200 ℃ for 5 min. Each deposition was repeated after 5, 10 and 15 times to increase the thickness. Next, the samples were annealed in a muffle furnace at 300 ℃ for 2 hours under oxygen atmosphere. Finally, a Finite Element Simulation was performed in order to obtain the amount of energy that this film would generate as a EH device.

3. Results and discussion



a) **b)** **c)** **d)** **e)**

Figure 1. Substrates of SS302 with 5 layers (a), 10 layers (b and c) and 15 layers (d and e). The substrates of 1b and 1d were subjected to annealed at x|300 °C for two hours.

The ZnO films was deposited on 2x2 cm SS302 steel substrates: one with 5 layers (Figure 1a), two with 10 layers (Fig 1b and 1c) and two more with 15 layers (Fig. 1d and 1e). A 10-layer substrate (Fig 1b) and a 15-layer substrate (Fig 1d) were annealed at 300 °C for two hours. To measure the thickness of the films, a step was made in each, using 1 molar hydrochloric acid (HCl) for 10 seconds. The films showed qualitative uniformity and adhesion, and in order to corroborate the presence of ZnO in the substrates of Figure 1, characterizations of percentage reflectance were performed. The reflectance percentage results (Figure 2) shows a first peak that starts at 370 nm. This confirms the presence of ZnO because it absorbs light with wavelength around 375 nm.

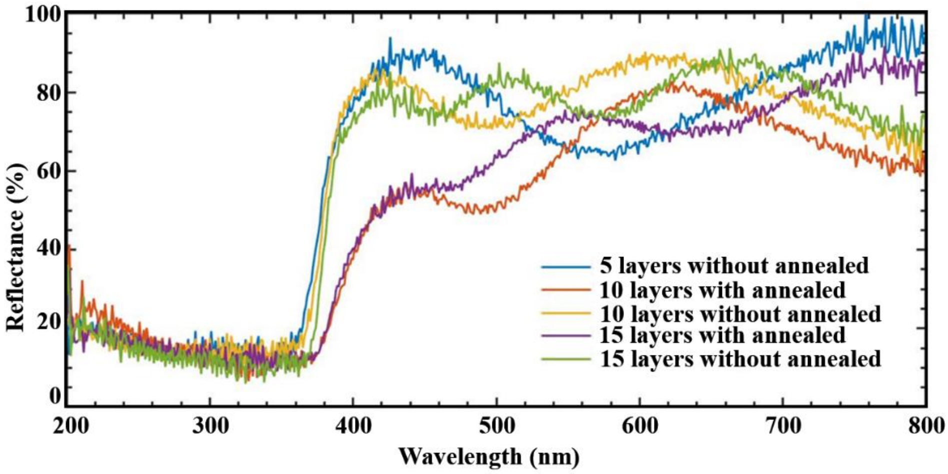


Figure 2. Results of reflectance percentage made to deposited films.

Also, figure 2 shows a sinusoidal oscillation in all measurements after the first crest, which indicates homogeneity in the films. In addition, it is observed that the measurements of the heat-treated films (Figure 1b and 1c) shows a shift to the right, indicating larger grain size in the film.

Table 1. Thickness of the different layers.

Deposition	Thickness (nm)
5 layers without annealed	972
10 layers with annealed	323
10 layers without annealed	593
15 layers with annealed	944
15 layers without annealed	1634

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Table 1 shows the thickness of the deposited layers obtained using a profilometer, showing that the thinnest layer was 972 nm and the thickest layer was 1634 nm. Figure 3 shows the results of the X-ray diffraction of the sample with 15 layers, observing peaks with 2Ө values at 25.29 °and 38.55°, it was possible to confirm the presence of ZnO with preferential orientation in the plane (002), as well as the stainless steel that acts as a substrate. Using a FEM software, the data obtained with the subtracted with 15 layers of ZnO was entered to obtain the voltage that would be generated by using it as EH. The SS302 substrate was drawn, posing as a cantilever beam. A seismic mass of neodymium was added with measures of 2x0.5x0.05 cm, this to decrease the resonance frequency of the device.

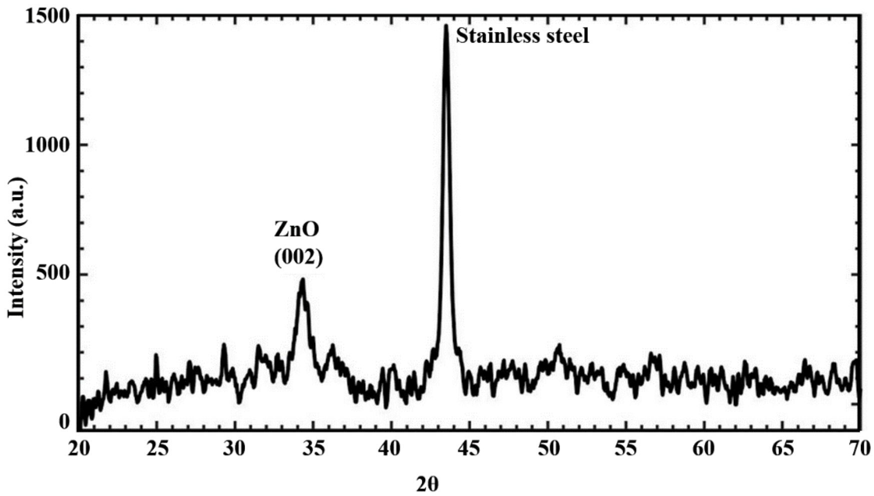

Figure 3. X-ray diffraction of the sample with 15 layers.

To solve the mathematical model, a mesh with 15090 elements was generated, having a greater refinement at the opposite end to the seismic mass as can be seen in Figure 4a. This is because is the area with the maximum strain when vibrations affect the device. Figure 4b shows the behavior of the stresses along the substrate in the first vibration mode, which is at 103.31 Hz.

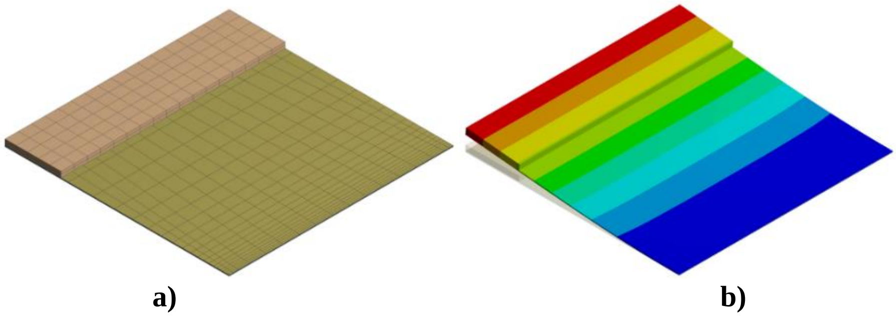

Figure 4. Figure 4a shows the mesh made to the geometry of the substrate with multilayer ZnO film. In the figure 4b shows the strain along the substrate when the device is excited at 103.31 Hz.

Figure 5 shows the graph that describes the voltage generated by the simulated EH, showing a sweep from 103 to 103.5 Hz, passing through the resonant frequency, where the highest voltage value is shown, this being 2.67 volts. As future work will be conducted electrical characterization to determine the voltage and power generated can obtain this device.

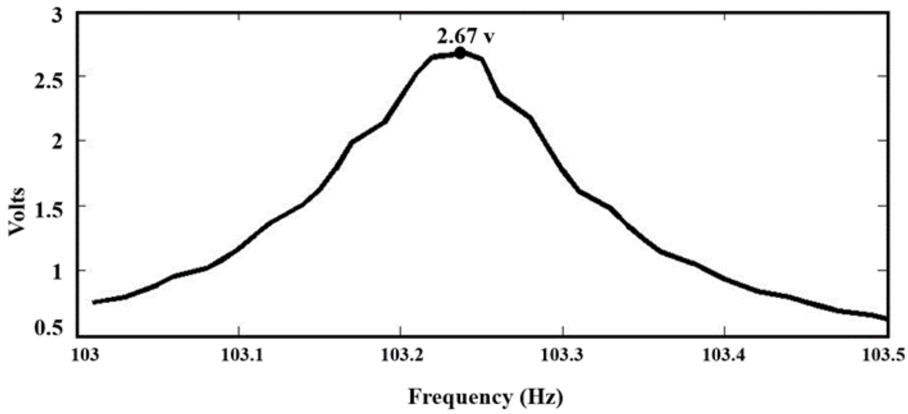

Figure 5. Graph of the frequency sweep to observe the generated voltage.

4. Conclusions

The depositions of ZnO films obtained on SS302 substrates were made using sol-gel method. With the spin coating technique, the number of deposited layers of 5, 10 and 15 layers was varied to increase the thickness of the films. The results of the XRD diffractogram and UV-Vis reflectance confirm the presence of ZnO, with homogeneous films in the preferential plane (002). These films can be used as energy harvesting microgenerators. The simulation performed shows that an EH with a seismic mass of neodymium with measurements of 2x0.5x0.05 cm could generate a potential difference of 2.67 volts when entering its first resonance mode at 103.31 Hz, thus functioning reusing the mechanical energy, like mechanical vibrations, in the environment surrounding the device. As a future work, the fabrication and characterization of the presented PEH will be performed, in order to compare the result obtained in the FEM simulation.

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