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The Effect of Annealing Temperature on Spin-Coated Zn-doped NiO Film for Humidity Sensing Applications

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Abstract* – Zinc (Zn)-doped nickel oxide (NiO) thin films were fabricated using spin coating technique for humidity sensing applications. Nickel acetate tetrahydrate was used as a precursor material and zinc acetate was used as the dopant source. The films were annealed at various annealing temperatures of 400 ºC, 500 ºC and 550 ºC in ambient. X-ray diffraction (XRD) was used to investigate the structural properties of the samples, while ultraviolet-visible spectroscopy (UV-Vis) was used to characterize the optical properties of the samples. The thickness of the samples was measured using a surface profiler which the thickness are 178.8 nm, 148.0 nm, and 116.9 nm for samples annealed at 400 ºC, 500 ºC and 550 º, respectively. The humidity sensing properties for each sample were characterized using humidity chamber equipped with humidity sensing measurement system which the relative humidity (RH) value was set between 40% RH and 90% RH. The XRD results showed that all samples were in the amorphous state. The average transmittance in the visible region obtained from the UV-vis spectra exceeded 90% transmission for all samples. The highest bandgap obtained is 3.6 eV for Zn-doped NiO annealed at 500 ºC. The humidity sensing results show that a high sensitivity sensor could be produced using sample annealed at 550 ºC.

Keywords: Zn doped NiO; spin coating technique; humidity sensing properties.

I. INTRODUCTION

Accurate humidity sensors particularly those that are prepared using facile and low-cost fabrication processes are important for humidity monitoring in several fields including medical, storage, semiconductor industries, agriculture, textile industries, and environment [1, 2]. The humidity sensing characteristics are analyzed through their signal change when the incidence of water vapour absorption occurs on the sensing surface. There are few types of techniques used to design humidity sensor which are resistivity, acoustic, mechanical, capacitive, and optical techniques. However, most of the research regarding the humidity sensor targeting a new material.

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Material engineering of the sensing layer, which focus on novel morphologies, also conducted to improve the humidity sensing properties and response time. Moisture and humidity are the terms that commonly used to represent water contents. It is important to precisely measure the water content in a different environment with different processes because even a small amount of water content can easily affect the physical and chemical nature of materials [3].

To date, the applications of high-quality humidity sensors are essential in various field. Humidity sensors are used in semiconductor industries to monitor the wafer processing areas [4]. Next, human breathing can be monitored by using humidity sensor in the medical sector [5-10]. By monitoring and detecting the abnormal respiratory rate, some major diseases could be diagnosed [11]. Some respiratory disorders and failures could be identified by monitoring breathing airflows such as apnoea and hypopnoea [12]. In agriculture sectors, the crop growth was monitored using humidity sensors [13]. The collected data from humidity sensors are directly transferred to the central control system via wireless network technology.

A reliable production method, low-cost preparation, and low-cost material but producing excellent humidity sensing performance are preferable for mass production. Considering these factors, this research provides a path towards achieving those components and at the same time improving the commercially available products. In this research, a simple method called a sol-gel spin coating technique was used to fabricate humidity sensors based on zinc (Zn) doped nickel oxide (NiO). Aside from low-temperature fabrication, these humidity sensors also operate well at room temperature without any external heating, which normally used to enhance humidity detection.

NiO materials have a wide range of energy bandgap from 3.6 eV to 4.0 eV with a simple cubic structure. Therefore, it is widely used in many applications such as transparent electrodes in photovoltaic devices. The advantages feature of NiO are it is a p-type and low-cost material. NiO also is known for its strong durability, large span optical density, promising ion storage materials in terms of cyclic stability, electrochemical stability, and many techniques can be used to manufacture NiO [14]. Due to NiO good responsivity, it can be used as a touch screen, photodetector, and it also can be used as a gas sensor because of its bandgap [15]. Other than that, NiO has great potential when it comes for applications in wide electromagnetics absorbers, packaging materials, wastewater catalyst, and batteries. These applications could be improved when the NiO doped with certain metals or it is mixed with metal oxides or polymer to produce composite. There are many reports available regarding lithium (Li) or cobalt (Co) doped NiO, however, there are less exploration on Zn-doped NiO [16]. The present study on Zn-doped NiO for humidity sensing can open a new insight of p-type material for humidity sensor applications.

It is proven that the sol-gel process is a useful approach for the researchers to prepare various materials. There are many experimental results showed that this method was suitable and successful to produce material on a large scale. Besides, the sol-gel method is frequently utilized to synthesize organic or inorganic hybrid nanocomposites as it can be performed under ambient condition [17]. The sol-gel method is an important process in semiconductor technology because it can produce high-quality thin films [18, 19]. This technology has excellent control of stoichiometry and composition modification, therefore it probably can offer to manufacture a large scale of high quality homogenous thin films using inexpensive equipment that can lead to a cost-effective process [19].

Herein, we prepared the Zn-doped NiO films via sol-gel spin-coating technique for humidity sensing applications. The prepared samples show promising results as its response well with the humidity.

II. Methodology

Preparation of Zn-doped NiO thin films was shown in Figure 1. The solution was deposited onto the glass substrate using the spin coating technique. The films were then undergone drying process at 250 ºC before it was annealed in a furnace at various temperatures of 400 ºC, 500 ºC, and 550 ºC. The thin films were then undergone the characterization process.

2.1 Solution preparation for Zn-doped NiO

Zn-doped NiO thin films were produced using the sol-gel spin coating method. First, the precursor solution was prepared using 2.4884g of nickel acetate powder, 0.1103g zinc acetate dihydrate powder and 2 ml diethanolamine in 50 ml ethylene glycol monoethyl ether. The prepared solution was then sonicated for half an hour at 50 ºC by using sonication bath. After the solution was sonicated, the hotplate magnetic stirrer was used to stir the solution for 1 hour at room temperature.

2.2 Deposition of Thin Film Using Spin Coating Technique

Spin coating method was used to deposit the thin films onto the glass substrate for each sample. The speed of spin coater (Laurell WS 650) was set at 4000 rpm for 60 seconds. The precursor was dropped onto the glass substrate using pipette during rotation of the spin coater. The sample was then undergone the drying process using a furnace for 5 minutes at 250 ºC. The dropping precursor and drying process were repeated 5 times to increase the thickness of the thin film.

2.3 Annealing Process

The samples were annealed at various temperatures of 400 ºC, 500 ºC, and 550 ºC using the furnace for two hours.

2.4 Metal Contact Deposition

Thermal evaporation technique (Ulvac-VPC-1100) was used to deposited silver (Ag) with a thickness of 60 nm as a metal contact on the glass substrate

2.5 Characterization Process

The crystallinity behaviour of each sample was characterized using X-ray diffraction (XRD, PANalytical X'Pert PRO). The optical properties of the samples were investigated using ultraviolet-visible (UV-Vis) spectroscopy (Jusco). Next, the samples were characterized using the humidity sensor measurement system to study the humidity sensing performance of the samples. In this measurement, a humidity chamber (Espec) was used with the sensor measurement unit (Keithley 2400) and software. The relative humidity (RH) in the humidity chamber was set between 40% RH to 90% RH. The thickness of the samples was measured using the surface profiler (KLA Tencor P-6).

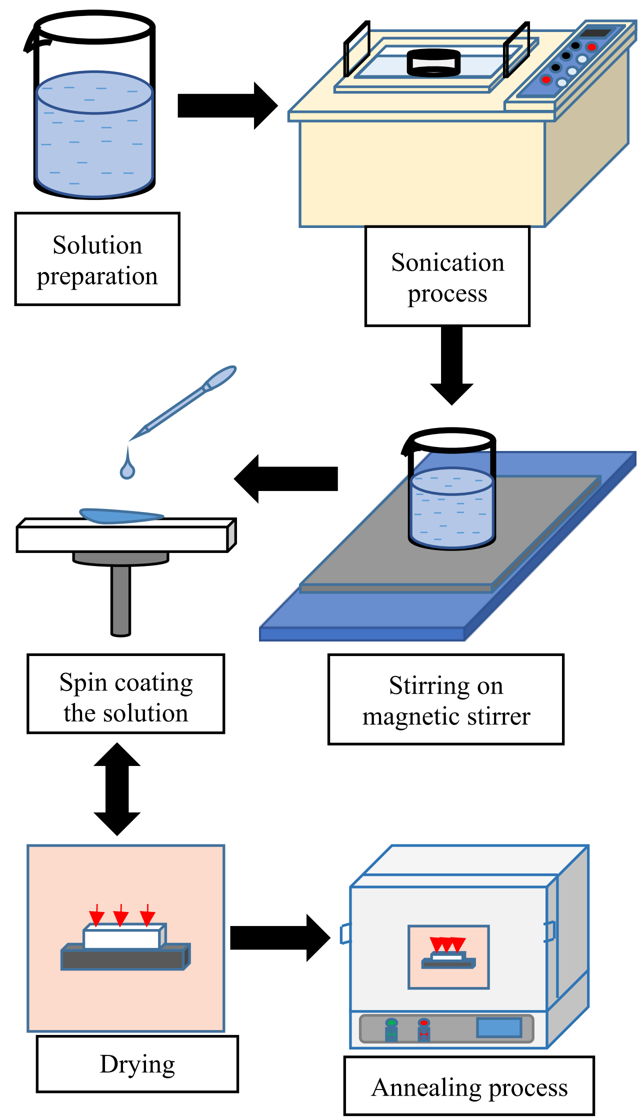

Fig. 1: Preparation of Zn-doped NiO thin films

III. RESULTS

3.1 Structural properties

XRD characterization was conducted to investigate the crystallinity of Zn-doped NiO at various annealing temperatures of 400 ºC, 500 ºC, and 550 ºC. From the XRD pattern in Figure 2, it can be seen that the XRD result for Zn-doped NiO films annealed at different temperatures have amorphous structures. There is only one peak appeared in the XRD pattern located at 42.6º, which corresponded to (200) plane. However, this peak exhibits broad and weak XRD intensity to indicate the poor crystallinity structure for all samples. This peak gradually diminished after annealed at high temperature, which might be due to the activation of dopant into NiO lattice. At high annealing temperature, the Zn dopant has sufficient energy to integrate into NiO lattice and creates structural defects, which transforms NiO into amorphous phase. This condition also occurs due to the crystallite structure of Zn-doped NiO is not well-built. It also could be observed in the XRD patterns for all samples that there is wide diffraction peak from 20º to 40º which represents the amorphous phase of the glass substrate [20, 21]. The result obtained from the XRD graph is contradicted with the study made by N. Parimon et al. [22] which reported that the peak of NiO is narrower when the annealing temperature increased. However, they studied on the nanostructured NiO, which prepared using immersion method and may have different NiO characteristics to that of the granular film prepared using sol-gel spin-coating method. In addition, the optimized annealing temperature for different materials might be varied depending on the annealing equipment, substrate, preparation process and others. Our results in Figure 2 shows that the high crystallization of Zn-doped NiO samples could not be detected and the films only exhibit amorphous characteristics. The amorphous structure or incomplete crystallinity of Zn-doped NiO occurs due to the appalling desorption of oxygen molecules in the precursor [23]. Martínez-Gil et al. prepared undoped NiO thin film using chemical water bath deposition at different annealing temperatures between 200 ºC to 400 ºC [24]. They found that the diffraction peak with very low intensity only appeared after annealing process at 400 ºC. However, the reduce in the peak intensity is commonly reported after doping with metal elements[25] .

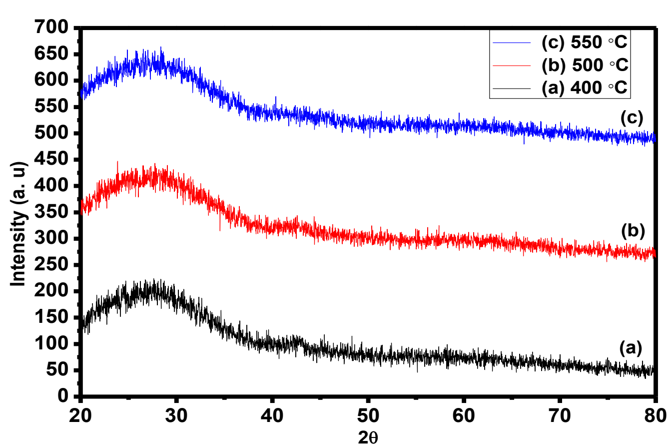

Fig. 2: XRD patterns for Zn-doped NiO at different annealing temperatures

3.2 Optical properties

The optical properties of Zn-doped NiO, which are characterized through transmittance and absorbance spectra in the wavelength regions between 300nm – 800nm is presented in Figures 3 and 4, respectively. The average transmittance of the samples was calculated between 400 nm and 800 nm and the data were tabulated in Table 1. Based on Table 1, the average transmittance values for all samples exceed 90%. Specifically, the transmittance values are 91.5%, 90.4% and 92.9% for Zn-doped NiO films at annealing temperatures of 400 ºC, 500 ºC, and 550 ºC respectively. These results suggested that the transmittance value for all samples are almost constant despite the difference in the annealing temperatures.

TABLE 1: AVERAGE TRANSMITTANCE AND BAND GAP VALUE FOR ZN-DOPED NIO AT DIFFERENT ANNEALING TEMPERATURES

Annealing temperature (ºC)	Average transmittance (%)	Bandgap (eV)
400	91.5	3.42
500	90.4	3.60
550	92.9	3.58

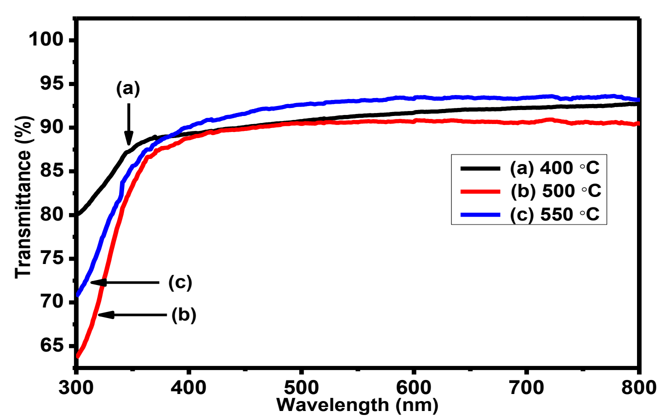

Fig. 3: Transmittance graph for Zn-doped NiO at different annealing temperatures

Figure 4 presents the absorbance spectra of Zn-doped NiO at annealing temperature varied at 400 ºC, 500 ºC, and 550 ºC. Higher absorbance value can be seen in the lower wavelength in Figure 4, which indicates that all samples exhibit high UV absorption at wavelength below 400 nm [26].

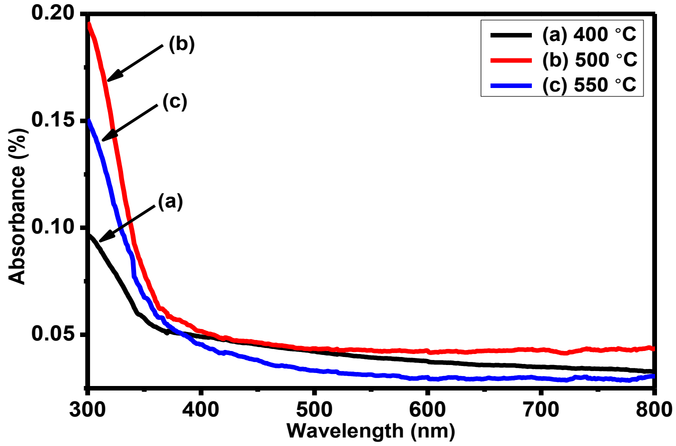

Fig. 4: The absorbance of Zn-doped NiO at different annealing temperatures

The thickness of thin films was measured using the surface profiler (KLA Tencor P-6). Figure 5 shows the thickness graph obtained from the surface profiler for the sample annealed at 400 ºC. The “St height” shown in Figure 5 indicates the thickness value of the film which is 178.8 nm. The thickness for the sample annealed at 500 ºC and 550 ºC are 148.0 nm and 116.9 nm respectively. The result shows that the thickness of the film reduces at higher annealing temperature due to densification of the grain.

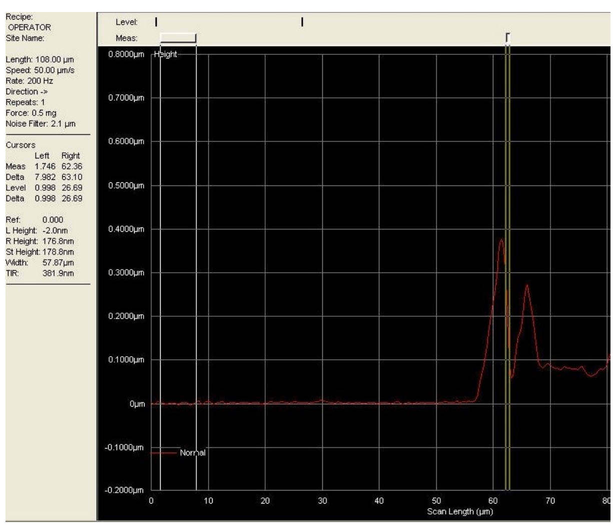
Fig. 5: Thickness measurement result for the sample annealed at 400 ºC

Next, the optical band gap values of the samples were calculated using Tauc’s plot relation based on the following equation (1):

$$\alpha h v = B(hv - E_g)^{1/2} \quad (1)$$

where E_g act as the optical band gap, B is constant, α is the absorption coefficient, and hv is photon energy. The Tauc’s plot of Zn doped NiO at different annealing temperatures of 400 ºC, 500 ºC, and 550 ºC are presented in Figure 6. The bandgap values were retrieved from the intercept at the x-axis of the plot and were tabulated in Table 1. The estimated values obtained from the intercept at the x-axis are 3.42 eV, 3.6 eV, and 3.58 eV for 400 ºC-, 500 ºC-, and 550 ºC-annealed Zn-doped NiO films, respectively.

Previous studies indicate that when annealing temperature increases, the value of the bandgap also increases [27, 28]. The bandgap values obtained from this study are still in good agreement with the reported band gap values of 3.15 – 3.80 eV for NiO thin films [29].

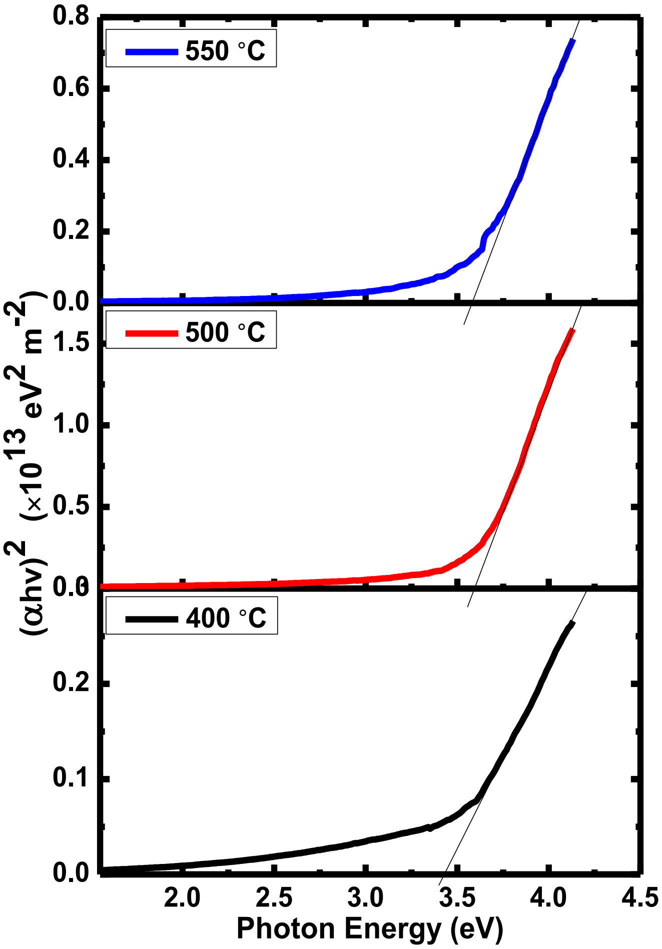

Fig. 6: Estimation of bandgap energy of Zn doped NiO at various annealing temperature by using Tauc's plot.

3.3 Humidity sensing properties

The humidity responses for Zn-doped NiO films were measured using the humidity sensor measurement system with humidity chamber. The sensing response represents by the current (I) versus time (t) plots, as shown in Figures 7. Measurements were conducted in a humidity chamber with the values of humidity level ranged between 40% RH to 90% RH. The bias voltage used to measure the humidity on the samples was fixed at 5 V in room temperature. The value of humidity level was changed from 40% RH to 90% RH and from 90% RH to 40% RH to characterize one cycle sensing response towards increasing and decreasing condition of relative humidity percentage.

The plot of humidity response at different humidity levels for Zn-doped NiO films annealed at temperatures of 400 ºC, 500 ºC, and 550 ºC are represented in Figure 7. The humidity response plot shows that all samples exhibit increased values of the current signal at the increasing RH values from 40% RH to 90% RH. The Zn-doped NiO films annealed at 500 ºC and 550 ºC show slightly stable current responses when the RH values were varied from 40% RH to 90% RH and show a smooth current response when the RH values decreased from 90% RH to 40% RH. However, the humidity sensing graph for 400 ºC-annealed film shows a slight deteriorated current signal at the increasing RH levels. This deteriorated signal for 400 ºC-annealed films is poorer than that of the response current signals of the samples annealed at 500 ºC and 550 ºC. The current signals for all samples gradually decrease when the humidity level is reduced from 90% RH to 40% RH. The humidity sensitivity was calculated for each sample to be 105.5, 45.8, and 107.7 for Zn-doped NiO annealed at 400 ºC, 500 ºC, and 550 ºC, respectively. The Zn-Doped NiO film annealed at 550 ºC shows the highest sensitivity followed by 400 ºC and 500 ºC-annealed samples. The sensitivity values of the humidity sensing were calculated using equation (2).

$$S = \frac{I_{90\%RH}}{I_{40\%RH}} \qquad (2)$$

Here, $I_{90\%RH}$ is the current value at 90%RH, while $I_{40\%RH}$ is the current value at 40%RH.

The humidity sensing plot also shows that the current signal for the samples annealed at 550 ºC is higher compared to other samples. This condition might be due to the surface of Zn-doped NiO film at an annealing temperature of 550 ºC can adsorb more water molecules, which enhances the movement of the ions in the film that converted into high current signals. Water molecules react with the sensors during the increased humidity levels through chemisorption, physisorption and capillary processes of the water molecules on the film's surface [30], in which the electrolytic and ionic conduction mechanisms are taking place. This mechanisms reduce the resistance of the sensor at the increasing humidity levels and contribute to the sensitivity of the sensor [26]. It should be noted that, the annealing temperature could be increased more if the expensive substrate such as alumina or pure silicon dioxide is used. The melting point of NiO is approximately 1,955 °C and therefore the study on annealing temperature below this point and their effects to the humidity sensing performance will provide a new insight into properties of NiO film.

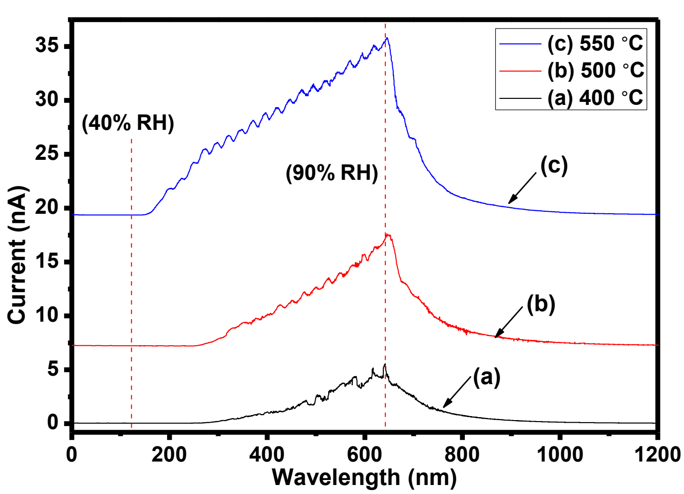

Fig. 7: Response curve of humidity sensing for Zn-doped NiO at different annealing temperatures

IV. CONCLUSION

Zn-doped NiO was successfully deposited using spin coating technique and annealed at different temperatures of 400 ºC, 500 ºC, and 550 ºC. The structural, optical and humidity sensing properties of the films were investigated. The XRD analysis showed that the prepared samples were amorphous NiO structures, with only one broad peak with very low intensity was observed in the XRD pattern. The bandgap of the Zn-doped NiO annealed at 400 ºC, 500 ºC and 550 ºC samples were 3.42, 3.60 and 3.58 respectively. The transmittance level for each sample was excellent with the average transmittance value exceeds 90% for every sample. All samples were responsive towards different range humidity level that varied from 40% RH to 90% RH. Among the samples, sample annealed at 550 ºC showed the highest sensitivity with the value of 107.7, followed by the samples annealed at 500 ºC and 400 ºC with sensitivity values of 105.5 and 45.8, respectively. These results suggested that the amorphous Zn-doped NiO thin films have high potential for humidity sensing application with good device performance.

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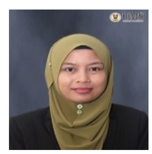

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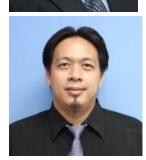

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