Influence of the distance between nozzle and substrate on the structural, photoluminescence, and detector characteristics of p-NiO/n-Si hetero-junction deposited by spray pyrolysis method

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Abstract

In the present investigation, p-NiO has been deposited on n-Si (100) substrate by the spray pyrolysis method. The effect of the distance between the substrate and the nozzle on the structural, photoluminescence, and detection properties has been well inspected. The highest film nucleation and the degree of crystallization were found at a distance between the nozzle and the substrate of 25 cm which is the preferred one that can affect the performance of the detector. The XRD analysis proved the polycrystalline system with a cubic structure for NiO. The FESEM analysis manifested nano and micro particles distributed on the Si layer, and the micro particles have porous like structures that play a significant role as photons guider. The photoluminescence measurement evinced three main peaks at the UV and visible regions of the electromagnetic spectrum, which is related to the near band edge emission and defects within the crystal, respectively. The I–V characteristics revealed a good conductivity under the UV illumination, and the highest current was recorded by a sample when the distance was 25 cm. The responsivity elucidated a high value at the UV region with 6.5 mA/W, and the current–time properties demonstrated good reproducibility, high stability and photoresponse, and rapid response, and recovery times of 0.375 s and 0.791 s, respectively at a lower bias voltage of 1.5 Volt under the UV photons source.

Keywords NiO · UV photodetector · Spray pyrolysis · Porous microstructures

1 Introduction

The enhancement of Ultra-Violet (UV) photodetectors is very significant; this is due to its employment in many fields, such as missile tracing, optical communication, chemical and biological sensors, image sensing, and flame detection (Chen et al. 2014a, b; Rajan et al. 2014). Many researchers have used metal oxide semiconductors due to their wide band...
gap, ease to fabricate, and low cost materials (Abed et al. 2019; Yousif et al. 2020; Abed et al. 2020). Some scientists have deposited these materials on Si, glass, alumina, sapphire, or quartz substrates and used them for various applications (Alwan et al. 2021a, b; Khudadad et al. 2021; Chala et al. 2018; Ozel et al. 2020). The enhancement process of UV photo detectors should involve response time, restoration time, responsivity, and detectivity (Alsultanay et al. 2016; Liu et al. 2020; Alam et al. 2020). For this purpose, numerous articles have tried to improve the performance of the detectors using metal oxide semiconductors, such as ZnO, TiO₂, NiO, SnO₂, andWO₃ (Ning et al. 2018; Chahrour et al. 2019; Parida et al. 2017; Marimuthu et al. 2019; Yadav et al. 2020). Among these materials, Nickel Oxide (NiO) is the preferred one; the reason behind the selection of this p-type metal oxide semiconductor is related to its direct and wide energy band gap, high surface area, high detectivity and responsivity, ease to fabricate, and low cost device (Gomaa et al. 2017, 2016; Singh et al. 2017). The perfect technique to improve the detector’s characteristics is the fabrication of P-N hetero-junction (Ji et al. 2020; Gunasekaran et al. 2020; Reddy et al. 2020; Thongma et al. 2019). The creation of p-Ni/n-Si hetero-junction leads to reduce the leakage current due to establishing a strong electric field at the interface between Ni and Si and in turns develops the photocurrent and the restoration time (Jayalakshmi et al. 2019). Therefore, it is significant to use this hetero-junction for detecting a wide range of UV and visible light in the spectrum region. Choi et al. (2005) evaporated NiO on a substrate of silicon, and their hetero-junction was responding to visible and UV regions. Zhang et al. (2014) fabricated a photodetector based on p-NiO/n-Si hetero-junction. This detector was created by UV oxidation of Ni, and it showed a low leakage of current density in comparison with that fabricated by the sputtering method. These detectors have a slow response time, low EQE, and low photo current and dark current, this is due to the amorphous structure of NiO which is reducing the lifetime of the carrier. There are several techniques for the NiO thin films deposition, such as pulsed laser deposition (Wang et al. 2015), spray pyrolysis technique (Krunks et al. 2014), chemical bath deposition (Sun et al. 2018), sputtering (Chen et al. 2014a, b), and spin coating method (Goumri-Said et al. 2019). Among these methods, the spray pyrolysis method is low cost, easy to prepare the film, high purity product, obtained homogeneous film, high chemical nucleation reaction, and thickness of the film well controlled through this method. According to an extensive search, few articles deal with the deposition of NiO by spray pyrolysis method on Si substrate and then study its detector characteristics. The aim of this work is to fabricate a UV detector based on the spray pyrolysis method of NiO on Si substrate and then study the effect of distance between the nozzle and the substrate on the structural, photoluminescence, and detector properties.

2 Experimental work

Before the deposition method, 1 cm² silicon substrates of (100) orientation, n-type, and 1–5 Ω·cm resistivity were well cleaned by washing them in ethanol and distilled water. After the cleaning, a spray pyrolysis system was provided for the deposition process, and it can be seen in (Habubi, Nadir F. et al. 2015). The starting material is 0.2 M of NiCl₂·2H₂O (99.9% purity from Changsha Easchem Co. Ltd., China), and this material was dissolved in 70 ml of 99.99% ethanol solution in a 100 ml beaker. Then, this solution was stirred into a magnetic stirrer for 25 min at the room temperature, and the obtained solution is greenish. The final solution was used in the spray pyrolysis system. The based conditions
of the spray process are as follows: the temperature of the deposition was fixed at 375 °C, the pressure of the nitrogen gas was fixed at 2.4 bar, the spraying time was 6 s. While, the duration between two sprays was 80 s to keep the temperature at its constant degree and to give the chance for more nucleation reactions, and the distance between the nozzle and the sample was varied (20, 25, and 30 cm) in order to select the optimized distance for the deposition process and its effect on the NiO/Si characteristics. Each film was deposited with 50 ml with the former solution.

3 Characterizations

The structural properties were investigated by X-ray diffractometer instrument (6000-SHIMADZU) with Cu Kα source (0.15406 nm wavelength), and the scanning 2θ is in the range of (30–70°). The surface morphology was inspected by field emission scanning electron microscope (FESEM) with a high resolution image (T EASCAN manufactured by UEGA. LM company, Geck origin), and the chemical element was confirmed by electron dispersive X-ray (EDX) analysis along with FESEM instrument. Photoluminescence (PL) (Hitachi 3100) with an excitation wavelength of 325 nm at the room temperature was used to investigate the emission spectra of the samples. The current–voltage characteristics of the hetero-junction were inspected by Hewlett Packard 34401A multimeter and Keithley 6517A Electrometer/High Resistance Meter with the voltage sweeps from −1.5 V to +1.5 V. The current–time properties were proved under the dark and illumination case using 365 nm LED at a constant voltage of 1.5 V. The thickness of the film was measured by a laser reflection method in which a 632 nm He–Ne laser was used, and the thickness of the films that deposited at 20, 25, and 30 cm distance were 463, 457, and 471, respectively. The schematic of the photodetector device and the electrode configuration are depicted in Fig. 1.

4 Results and discussion

4.1 XRD analysis

The purpose of using X-Ray diffract-meter analysis is to identify the employed materials in the work, and after confirming the materials, one can determine the other related parameters. The XRD patterns of NiO/Si at different distances between the nozzle and the substrate have been investigated and depicted in Fig. 2. In this figure, it could be observed that there are three main peaks at 2 thetas of 37.28, 43.38, and 62.98° which are corresponding to the main planes of (111), (200), and (220), respectively. All of the patterns showed a
polycrystalline system with a cubic structure. Also, from the figure, one can notice that the intensity of the detected peaks has increased when the distance between the nozzle and the substrate increases from 20 to 25 cm, whereas it has decreased when the distance increases to 30 cm. The obtained patterns are matched with those previously reported (Fasaki et al. 2010; Gokul et al. 2013; Arif et al. 2016; Yousaf et al. 2020; Chtouki et al. 2017). The increase in intensity leads to a high degree of crystallinity. The crystal size, dislocation density, and micro strain were calculated and tabulated in Table 1. They were calculated by the following formulas (Alwan et al. 2019; Othman et al. 2017; Yousif and Khudadad 2020):

\[
D = \frac{k \times \lambda}{\beta \times \cos \theta}
\]  
(1)

\[
\delta = \frac{1}{D^2}
\]  
(2)

\[
\text{Micro strain} = \frac{\beta \cos \theta}{4}
\]  
(3)

where \( k \) is the shape factor with a value of 0.9, \( \lambda \) is the wavelength of the radiation, \( \beta \) is the full width at half maximum, and \( \theta \) is Bragg’s angle.

Table 1 XRD parameters of NiO/Si at different distances between the nozzle and the Si substrate

| Distance between nozzle and substrate (cm) | 20 of (111) main plane (deg.) | FWHM (deg.) | Crystal size (nm) | Dislocation density (nm\(^{-2}\)) | Micro strain |
|-----------------------------------------|--------------------------------|--------------|------------------|---------------------------------|--------------|
| 20                                      | 37.28                          | 0.3          | 27.94            | 0.1891                          | 0.00124      |
| 25                                      | 37.28                          | 0.22         | 38.1             | 0.1619                          | 0.00091      |
| 30                                      | 37.28                          | 0.26         | 32.24            | 0.1761                          | 0.00107      |
From Table 1, it is observed that the crystal size increased when the distance between the substrate and the nozzle has increased from 20 to 25 cm. This leads to high crystallinity and nucleation processes. While, it decreased when the distance has increased to 30 cm. Moreover, the dislocation density and the micro strain decreased, and the optimum value were found at the distance of 25 cm, in which the defects have decreased. The crystal size and the dislocation density have been well monitored, and the reason behind this trend is linked to the distance between the nozzle and the substrate which is the main factor that impacts the size of the crystals and hence improves the degree of crystallization. In the case of the distance 20 cm, the droplet is large, and when it reaches the substrate, there is not enough temperature to vaporize the droplet; hence it will be a weak cohesion of the film on the substrate. In the case of 30 cm, the droplet is small and it is vaporized before it reaches the substrate, hence when it touches the substrate, it will transform to powder. Whereas in the case of 25 cm, the droplets with an optimum size reach the substrate and evaporate at the moment before they touch the substrate, and then it will be a good film with a high cohesion with the substrate (Alwan et al. 2021a, b; Yousif et al. 2021). Therefore, it is very important to adjust the distance between the nozzle and the substrate.

4.2 FESEM analysis

The suitable method to analyze the surface morphology of thin film is the field emission scanning electron microscopy (FESEM) in which the beam of electrons is accelerated with a high voltage toward the film. The interaction between the accelerated electrons and the electrons of the target materials leads to the scattering of electrons, and the scattered electrons will be detected by a detector that will plot the morphology of the target materials. The surface morphology of Ni deposited on the Si substrate at different distances between the nozzle and the substrate is illustrated in Fig. 3. Figure 3a evinces the morphology of Ni/Si deposited at a distance of 20 cm, also this figure depicts the microstructure particles and the nanostructure particles below them. These particles covered the silicon layer, and this is due to the small distance between the nozzle and the substrate that makes the particles aggregated and covered the substrate. Figure 3b (the distance between the nozzle and the substrate is 25 cm), manifests the porous microstructure particles which not cover the whole silicon layer, there are spaces between these microstructures that contain small nanoparticles, and the parts of the silicon layer are exposed. These large porous like structures in the microparticles will be a good guide for the incident photons in order to enhance the detector performance. When the distance between the nozzle and the substrate is 30 cm, as in Fig. 3c, it demonstrates the large exposure areas (not covered). Furthermore, on the right side of the figure, there is a big and bright microstructure, the brightness is due to the high resistance of the target material which will slow the speed of the accelerated electrons, then these electrons will interact with the other accelerated beam, and this interaction will appear as a high brightness. The thickness of the film that deposited at a distance of 20 cm is 463 nm which is related to the small and large particles that packed on each other. For the film that deposited at a distance of 25 cm, the thickness of film is 457 nm, the decrement in thickness can be linked to the droplet size being smaller, and hence the particles are packed beside each other. While the thickness of the film that deposited at a distance of 30 cm is 471 nm, this increment is attributed to the transforming of the droplet to powder as mentioned in the XRD section, and then some particles will be stacked on each other. Generally, the morphology of the film is one of the most important parameters
Fig. 3 FESEM images of NiO deposited on Si at different distances between the nozzle and the substrate

that can affect the application under study (Zan et al. 2021a, b; Zan et al. 2021a, b; Chai et al. 2021). Some articles have also obtained the microstructure particles of NiO. (Manikandan et al. 2013) found the agglomerated NiO microstructures flaks. (Zhao
et al. 2011) determined the micro spherical NiO particles, mesoporous cubic, and hexagonal nanocrystal. (Xu et al. 2014) obtained the micro platelets morphology. (Nabi et al. 2020) found the Vanadium doped Ni microstructures of random shapes. (Tamura et al. 2017) determined the micropatterns structures of NiO/Cr fabricated by femtosecond laser reductive.

The particles distribution can be seen in Fig. 4, and this figure confirms the Gaussian distribution of the NiO microstructure particles. The particles distribution of the sample that deposited at a distance of 20 cm is in the range of 1–10 μm. After the distance is increased to 25 cm, the range of particles distribution is 1–20 µm. While for the distance of 30 cm, the range extended from 1 µm to 30 µm. Also, there are fewer particles having diameters of 30–41 µm.

4.3 EDX analysis

This measurement is typically used to specify the chemical elements and their weights. The chemical element analysis of Ni/Si that deposited at different distances between the nozzle and the substrate is shown in Fig. 5. This figure confirms the presence of Ni, O, and Si chemical elements without any other unwanted materials. When the distance between the nozzle and the substrate is increased from 20 to 25 cm, as in Fig. 5 a and b, the weight of the Ni element increased by about 3%, the weight of the O element decreased by about 5%, and the weight of Si element increased by about 2%. The rising in the weight of the Ni element leads to a high crystallization process, while the reduction in the weight of the O element is related to the reduction in defects (as proved from XRD analysis), and the rising in weight of the Si element is linked to the coalescence of nanoparticles and gave the opportunity for the silicon layer to appear in some regions. Whereas, the film that deposited on the Si substrate by a nozzle with a distance of 30 cm from the substrate (Fig. 5c) demonstrates the lowest percentage of Ni element, a higher percentage of O element than that in Fig. 5b, and the highest percentage of Si element.
Fig. 5  EDX for the chemical elemental analysis for NiO deposited at different distances between the nozzle and the Si substrate: a 20 cm, b 25 cm, and c 30 cm
4.4 Photoluminescence

The photoluminescence of NiO/Si was tested at the room temperature with an excitation wavelength source of 325 nm. Figure 6 reveals the PL spectra of the NiO deposited on the Si substrate at various distances between the substrate and the nozzle. This figure reveals that there are three main peaks related to the UV and Visible regions of the electromagnetic spectrum. The peak at the UV zone is linked to the near band edge emission of NiO which is influenced by the arrangement of the stoichiometric of anions and cations. This peak is located at around 366.68 nm for the distances of 20 and 30 cm, which is corresponding to 3.38 Ev of energy band, while it is located at 368 nm for the distance of 25 cm, which is corresponding to 3.36 Ev. The other peak is positioned in the visible zone that is connected to the defects within the NiO crystal, such as oxygen vacancies, oxygen interstitial, and Ni interstitial and these defects could act as the trappers for the electrons (Majumder et al. 2016; Jayalakshmi et al. 2018).

The other two peaks are located at 442 nm and 532 nm (visible region) for the films that deposited on a silicon substrate with the distances 20 and 30 cm, respectively, and these peaks are corresponding to 2.8 and 2.33 Ev, correspondingly. However, the peaks of the film that deposited on the silicon substrate with a distance of 25 cm are located at 446 and 534 nm which are corresponding to 2.78 and 2.32 Ev, respectively.

From the above description, one can note that there is a small shift towards the high value of wavelength when the film is deposited with a distance of 25 cm, and this is related to the slight increase in the grain size of the structure.

4.5 Current–voltage characteristics

The current–voltage characteristics of p-NiO/n-Si are portrayed in Fig. 7. This detector was tested under the dark and was illuminated with a blue light source (Light Emit Diode) with a power of 2 mW and 365 nm wavelength. The performance of the p-Ni/n-Si in the dark case shows a rectifying trend which improves the creation of the p–n junction at the interface between the p-Ni and the n-Si, as in Fig. 7a. The photodetector

Fig. 6 PL spectra of the NiO deposited on the Si substrate at different distances
depicted an improved photocurrent under the illumination of the blue light source. In this case, the blue light motivates the material to generate electron–hole pairs. At the same time, the electric field has been built at the interface by the applied reverse bias voltage, which serves as an electromotive force to transfer the electron to the silicon region and transfer the holes to the Ni region, Fig. 7b. Furthermore, it can be noticed that when the Si substrate was sprayed by Ni at a distance of 25 cm, it showed a good rectifying behavior under the dark and illumination conditions. This is related to the large width of the depletion layer, large grain size, and a high degree of stoichiometry (Mousa et al. 2019), also, the increase of crystal size gives a chance to the reduction of the scattering process by charge carriers (Khayatian et al. 2016). Moreover, the rising of photocurrent with the augmentation of voltage value is due to the increasing in the carrier drift velocity (Soci et al. 2007).
4.6 Detector properties

The responsivity of Ni/Si at a bias voltage of 1.5 V and a wavelength range of 300–900 nm at a constant power of 2 mW for the sample that deposited at a 25 cm distance between the nozzle and the substrate is exhibited in Fig. 8. There are two main peaks for responsivity, one of them is sharp, high intensity, in the UV-region of the electromagnetic spectrum, and the responsivity is around 6.5 mA/W. The other peak is broad, low intensity, and at the NIR region of the electromagnetic spectrum, as well as the responsivity is around (3.4 mA/W). The reason behind the presence of two regions for the responsivity is related to the existence of two materials Ni and Si, the Ni is the response to the UV light, while the Si is the response to the NIR light. Subsequently, the higher responsivity at the UV region is linked to the energy gap of the NiO which is near to this wavelength, while the lower responsivity is related to the energy gap of the Si layer which is matched to the NIR wavelength. When the energy of the incident photons was lower than the energy gap of the NiO material, the photons will pass through the NiO and be absorbed by the silicon layer which will produce electron–hole pairs in the silicon layer. In this situation, the transport of electron–hole pairs will be affected by the built-in electric field which exists at the interface between Ni and Si. Hence, the obtained device can operate in two regions, but it is effective for the UV region detectors. At the visible region, the responsivity decreased, and this is due to that there is not sufficient energy to establish electron–hole pairs. The responsivity was calculated by the following equation (Jwied et al. 2021):

\[
\text{Responsivity} = \frac{I_{ph} - I_d}{P_{op}}
\]

where, \(I_{ph}\) is the current under the illumination case, \(I_d\) is the current under the dark case, and \(P_{op}\) is the optical power of the photon source.

The most important factors for the photodetector device are the repeatability and the speed of detection. Figure 9 demonstrates the photocurrent as a function of time at a reverse bias of −1.5 V for the Ni sample that deposited on the Si substrate at a distance of
The switching test was tested under a 365 nm wavelength LED source. The trend of this measurement shows a good reproducibility and almost a high stability for the UV detector device. It can be observed from the figure that the five-cycle has a rapid response time when the UV light is switched on and also a rapid recovery time when the light is switched off.

The response and recovery times of the Ni/Si device are based on the oxygen adsorption and desorption rate process. Before the illumination (dark case), the oxygen molecules were adsorbed on the surface of the sample (Cheng et al. 2008). These molecules will restrict the free electrons, hence the oxygen species will form (Azimirad et al. 2014; Lim et al. 2010). According to this process, the conductivity decreased, and the depletion zone was established near the surface (Kar et al. 2009). The lowest conductivity can result from the development of a high amount of oxygen species on the surface of the material which extends the band bending (Dhara et al. 2011). The second step is when the material is illuminated by UV light which has energy higher than the band gap of the material; this leads to the generation of electron–hole pairs. Then, by combining the holes created by oxygen species on the surface, the oxygen species were released (Chen et al. 2010). At the same moment, the free electrons will accumulate on the electrodes. Therefore, the life time of the created electrons will rise (Safa et al. 2018). Because Ni material has hole carriers, the conductivity will rise (Chu et al. 2020). The main reason behind the high photoresponse and quick response and recovery times is connected to the porous-like structure which will make the detector device able to adsorb more oxygen molecules (as shown from FESEM for the sample that deposited with a 25 cm distance between the nozzle and the substrate), and also the presence of nanoparticles beside these microstructures made the material possess a high surface area. These two effective parameters act as an anti-reflective surface which guides the photons to generate a high amount of electron–hole pairs (Tsai et al. 2011).

Figure 10 reveals an enlarged image of one cycle from Fig. 9, and that presents the response and the recovery times when the UV light switched ON and OFF, respectively. The response time (τ_res.) is the duration that is required to increase the current from 10 to 90% before it reaches the saturation point. Whereas, the recovery time (τ_rec.) is the time
that the current is required to decrease from 90 to 10% before it reaches the reference point. As appeared from the figure, the response time is 0.375 s, and the recovery time is 0.791 s. In order to manifest the contrast between the current work and the other previous works, Table 2 lists the comparison of the performance of the photodetector based on the NiO material.

5 Conclusion

Nickel Oxide material was sprayed on n-(100) silicon substrate by spray pyrolysis method successfully. The effect of the distance between the nozzle and the substrate has been well studied. At a distance of 25 cm, the particles have been well arranged with little particles agglomerations; also the pores with large diameters have appeared which are acting as a guide for the photons. XRD and EDX analyses confirmed the presence of NiO and
| Detector                  | Method of deposition | Morphology                                      | λ (nm) | Power density (mW/cm²) | Bias voltage (V) | Response time (s) | Recovery time (s) | Responsivity       | Ref                      |
|--------------------------|----------------------|-------------------------------------------------|--------|------------------------|------------------|-------------------|--------------------|---------------------|-------------------------|
| p-PNZO/p-NiO/n-AZO       | Spray Pyrolysis method | –                                               | 365    | 4                      | 3                | 10.8              | 8.4                | 5.53 (A/W)          | Amiruddin, Kumar (2016) |
| TiO₂/NiO                 | Hydrothermal method   | TiO₂ nanowell/NiO mesoporous nanosheet          | 350    | 1.2                    | 0                | 12                | 7.1                | 0.042 (A/W)         | Zheng, et al. (2016)    |
| NiO/ZnO                  | Electrosprun hydrothermal | nanofiber                                     | 350    | 75 W                   | 0                | 7.5               | 4.8                | 0.415 mA/W          | Zhang et al. (2019)     |
| p-NiO/n-Si               | Hydrothermal          | nanoflakes                                     | 450    | 2.5 mW                 | −3               | 6.18              | 1.83               | –                   | Jayalakshmi et al. (2019) |
| NiO/Si                   | Spin coating          | nanosheets                                     | 385    | 1.1                    | −1               | 0.5               | 0.526              | 156.3 µA/W          | Parida et al. (2017)    |
| n-Si (111)/p-NiO         | Hydrothermal          | nanosheets                                     | 350–600 nm | 0.5            | −2               | < 30 ms            | < 30 ms            | 0.43 mA/W           | Zhang et al. (2017)     |
| p-NiO/n-Si               | Sputtering            | nanoparticles                                   | 318    | −                      | −0.5             | −                 | −                  | 4.8 A/W             | Hammadi et al. (2015)  |
| p-NiO/n-Si (100)         | Spray pyrolysis       | Mixed of nanoparticles and porous microstructures | 365    | 2                      | −1.5             | 0.375             | 0.791              | 6.5 mA/W            | Current work            |
Si without any other unwanted materials. A high degree of crystallinity and stoichiometry has been obtained for the sample with a 25 cm distance, as proved by the reduction of oxygen element from the EDX analysis. Also, this sample evinced a surface with a mixture of micro and nano particles, and the microparticles have a porous-like structure. The photoluminescence elucidated spectra that have three main peaks lie in the UV and visible regions of the electromagnetic spectrum which confirmed the near band edge emission and the existence of the defects, respectively. The current–voltage characteristics depicted well rectifying properties with a higher photoresponse under 365 nm compared with the dark current. The photoresponse of this device is connected to the movement of electrons to the n-Silicon and holes to p-Nickel Oxide by a high built-in electric field. Furthermore, the current–time characteristics revealed the swift response and recovery times toward the UV light with a constant reproducibility and a high stability. The tested detector has been done under a lower bias voltage of 1.5 Volt. Subsequently, the obtained device can be used in the optoelectronic detectors.

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Declarations

Conflict of interest The authors have not disclosed any conflict of interest.

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