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Investigation of NiO film by sparking method under a magnetic field and NiO/ZnO heterojunction

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Abstract

Nickel oxide (NiO) film receives attention from the field of optoelectronics due to its wide band gap and high transparency. By using a sparking method, the deposition of the NiO film is facile and unique. However, the NiO film made by the sparking method indicates a porous surface with an agglomeration of its particles. In order to reduce the porosity of the NiO film, the assistance of a permanent magnet in the sparking apparatus is presented. Here, we report the investigation of the NiO film and the p-NiO/n-ZnO heterojunction deposited by the sparking method under a magnetic field. Our results demonstrate that the porosity of the NiO film was reduced by increasing the magnitude of a magnetic field from 0 mT to 375 mT. Furthermore, the crystallinity and the electrical properties of the NiO film are improved by the influence of a magnetic field. For heterojunction, the best device shows the rectification ratio of 95 and the ideality factor of 4.92. This work provides an alternative method for the deposition of the NiO film with promising applications in optoelectronic devices.

1. Introduction

The p-n heterojunctions of nickel oxide (NiO) and zinc oxide (ZnO) films have many potential applications for optoelectronic and have been intensively studied due to their wide band gap [1–5]. NiO, an intrinsic p-type semiconductor with a band gap of ~3.6 eV, has been used as a transparent conducting oxide and hole transport layer [2, 4, 5]. In parallel, ZnO displays the n-type semiconductor with a direct band gap of ~3.3 eV, which is suitable for the electron transport layer of a solar cell [1, 3]. Currently, ZnO films are deposited by using the spin coating and spray pyrolysis methods, which consist of inexpensive and non-complex apparatus [6, 7]. In contrast, the NiO films by these methods not only require a toxic precursor but also produce the porous film [8, 9]. Presently, the NiO films are synthesized by chemical vapor deposition (CVD), electron beam evaporation and sputtering. These methods produce the high-quality and homogenous of the NiO film, but nevertheless requires the complex apparatus and vacuum atmosphere [10–12]. Therefore, an alternative method for synthesis of NiO film using a non-complex system and eco-friendly was sought out.

The sparking method, in addition to a non-complex method, is equipped with an inexpensive apparatus, operating without a vacuum pump and well applied with various substrates [13, 14]. Recently, it has been reported that the NiO thin film was successfully deposited by the sparking method [15]. Meanwhile, the addition of a magnet below the substrate in the sparking discharge apparatus was introduced by Ručman et al [16]. Their report revealed positive results as regards the crystallization of NiO film due to the influence of the magnetic field. However, NiO film made by the sparking method has a porous surface due to the agglomeration of its particles [15, 16]. In this study, we aim to reduce the porosity of the NiO film by placing permanent magnets...
above and below the substrate of the sparking method. This strategy helps to create a uniform magnetic flux, causing the uniform alignment of particles, which reduces the gaps at the surface of the NiO film [17].

In this work, the NiO film was deposited by using the sparking method under the magnetic field. It was found that the porosity of the NiO film decreased with the increase in the magnitude of a magnetic field. Furthermore, the magnetic fields also improved the crystallinity and reduced the dislocation density of the NiO film. The investigation of the optical properties of the NiO films indicated that the band gap decreased while the magnitude of a magnetic field increased. The capability of the NiO film in electronic applications was characterized by the current-voltage (I-V) curve measurement of the NiO/ZnO heterojunction. The results revealed that the NiO/ZnO heterojunction exhibited non-linear curves. Moreover, the increase in a magnetic field helped to improve the ideality factor as well as the rectification ratio of the NiO film. Based on these results, the assistance of a magnetic field in the sparking method is a promising way to modify the properties of the NiO film.

2. Experimental

2.1. Sol-gel synthesis of ZnO

The chemicals in this research were supplied by Sigma-Aldrich. The sol-gel of ZnO was synthesized by mixing 4.4 g of zinc acetate dehydrate with 100 ml of ethanol. The solution was stirred on a hot plate magnetic stirrer at 60 °C for 2 h. After that, 1.2 ml of monoethanolamine (MEA) was added to the mixed solution and stirred at 60 °C for 2 h. A transparent solution was achieved. Then it was aged at room temperature for 24 h before using for deposition [8].

2.2. Preparation of the sparking apparatus

As shown in figure 1(a), nickel wires (99.98%, Nano Generation) with a diameter of 0.5 mm were used as anodes and cathodes for the sparking method. The distance between the electrodes was 1 mm. The voltage of a high voltage DC generator was approximately 3 kV. The distance between the nickel wires and a substrate was 5 mm. The pulse frequency of the sparking was 13 Hz. Note that the sparking method was operated under ambient air and the output power of the sparking method was approximately 35 watt, which is high enough to break a double bond of oxygen gas but not high enough to break a triple bond of nitrogen gas.

2.3. Motion of charged particles in a magnetic field

In order to identify the motion of charged particles in a constant magnetic field, we used the Lorentz force (F) determined by the following equation [18]:

\[ F = qvB \sin \theta \]

where \( v \) is the particle velocity, \( q \) is the elementary charge, \( B \) is the magnetic field and \( \theta \) is the angle between \( B \) and \( v \). By deriving from (1) with the \( \theta \) equal to 90°, the motion of charged particles in a constant magnetic field is a helical as shown in figure 1(a).

2.4. Fabrication of ITO/ZnO/NiO/Ag

The 1 x 1 cm ITO glass was cleaned by sonication in distilled water, acetone and ethanol, respectively. Subsequently, the ITO glass was dried by N2 gas. The sol-gel of ZnO was coated onto the ITO by spin coating at 2000 rpm for 30 s. Then, the ITO/ZnO was annealed at 425 °C for 2 h under ambient air in a furnace. Next, Ni was deposited on the ITO/ZnO by sparking method for 30 min under the magnetic fields of 0 mT, 125 mT, 250 mT and 375 mT, respectively. Note that the magnetic field increased when increasing the number of permanent magnets. After deposition, the sample was annealed at 425 °C for 2 h under ambient air. Subsequently, the ITO/ZnO/NiO was covered with silver paint to create a top contract, which shows in figure 1(b).

2.5. Characterization

The samples were characterized by a scanning electron microscope (SEM, JSM-6335F), x-ray diffraction spectrometer (XRD, Philips X’Pert MPD), UV–vis spectrophotometer (Cary 50), ellipsometer (alpha-SE), X-ray photoelectron spectrometer (AXIS ULTRADLD XPS, Kratos analytical, UK) and source measure unit (Keithley 2450), respectively.

3. Results and discussions

The SEM images of NiO films by sparking method under the magnetic fields of 0 mT, 125 mT, 250 mT and 375 mT are given in figure 2. The morphology of 0 mT in figure 2(a) illustrates the porous surface, containing an
agglomeration of particles. By adding the magnetic field in the sparking method, the NiO films become denser and exhibit lower porosity as shown in figures 2(b)–(d). This result corresponds to the increase in an ion lifetime and the decrease in a scattering of charged particles induced by the magnetic flux \[19\]. Therefore, the particles are arrayed in an orderly manner and packed more tightly. However, too much magnitude of a magnetic field (375 mT) leads to the formation of nanoparticles at the surface of the NiO films as shown in figure 2(d).

The XRD patterns of ZnO, 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO are shown in figure 3. The results of an investigation indicate that the XRD pattern of ZnO consisted of 3 peaks, which associates with the JCPDS card no. 36-1451. For 0 mT NiO, the XRD pattern did not display any peaks except the pattern of a glass substrate, which confirmed that the structure of 0 mT NiO was amorphous. Meanwhile, 125 mT NiO, 250 mT NiO and 375 mT NiO indicate the prominent peak at 43.32°. This peak is correlated to the (200) plane with the d-spacing of 0.209 nm. Moreover, it has been observed that the increase in the magnetic field indicated a stronger intensity of (200) peak and illustrated a better crystal structure due to uniform alignment of particles induced by the magnetic flux. In order to further investigate the crystallinity of the NiO films, the following equations were used \[20, 21\]:

\[
D = \frac{0.9\lambda}{\beta \cos \theta}
\]  

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

where \(D\) is the crystallite size, \(\lambda\) is the wavelength of x-ray \(1.5418 \text{ Å}\), \(\beta\) is the full width at half maximum (FWHM) of the diffraction peak, \(\theta\) is Bragg angle of the diffraction peak and \(\delta\) is the dislocation density. The \(D\) of 125 mT NiO, 250 mT NiO and 375 mT NiO is calculated to be 30.7 nm, 35.6 nm and 40.7 nm, respectively. By deriving from (3), the \(\delta\) of 125 mT NiO, 250 mT NiO and 375 mT NiO is approximately \(10.6 \times 10^{14}\) lines \(\text{m}^{-2}\).
7.9 \times 10^{14} \text{ lines m}^{-2} \text{ and } 6 \times 10^{14} \text{ lines m}^{-2}, \text{ respectively. As a result of the increase in the magnetic field, the crystallite size of the NiO film increases while the dislocation density decreases.}

The transmission spectra are shown in figure 4(a). It was observed that the average transmittances (wavelength of 400–700 nm) of ZnO, 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO were 99%, 95%, 92%, 81% and 72%, respectively. By using the ellipsometer, the thickness of ZnO, 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO was measured to be 104 nm, 107 nm, 416 nm, 836 nm and 958 nm, respectively. This result demonstrates that the increase in the magnetic field strength helps to collect more particles, resulting in an increase in the thickness of the NiO film. Figure 4(b) displays the absorption spectra of the NiO films. The more the magnitude of a magnetic field adds, the more absorption intensity increase. This result corresponds to the increase in thickness of NiO films measured by ellipsometer. The absorption edge of the NiO films indicates the red shifts with the increasing of a magnetic field, reflecting a decrease in the band gap of the NiO films. To further verify the optical band gaps of the samples, Tauc’s relation were used [22]:

![Figure 2. SEM images of (a) 0 mT NiO, (b) 125 mT NiO, (c) 250 mT NiO and (d) 375 mT NiO after annealing.](image)

![Figure 3. XRD spectra of ZnO, 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO films on glass substrate.](image)
where $h$ is Planck’s constant, $\nu$ is the photon’s frequency, $A$ is a proportionality constant, $E_g$ is the band gap, $\alpha$ is the absorption coefficient and $n$ is equal to 2 or 1/2 for indirect and direct transitions, respectively. The $\alpha$ of the samples is expressed as [23]:

$$\alpha = 2.303 \left( \frac{A_b}{t} \right)^n$$

where $A_b$ is the absorbance and $t$ is the thickness of the samples. The plot $(\alpha h \nu)^2$ versus $h \nu$ is displayed in figure 4(c), showing that the optical band gaps of ZnO, 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO are found to be 3.21 eV, 3.51 eV, 2.79 eV, 2.45 eV and 2.19 eV, respectively. The decrease in the optical band gap of the NiO is originated by the increase in crystallite size and the transition from amorphous to crystalline. This result is in agreement with absorbance and XRD. In addition, the result is also correlated to other metal oxides reported by Ramana et al and Shariffudin et al [24, 25].

The chemical state of NiO film is analyzed by using XPS. The results of an analysis are displayed in figure 5 and table 1. As shown in figure 5(a), the survey spectra of NiO film confirm the presence of Ni, O and C without other elements, which prove that the NiO films are pure. The existence of carbon in the NiO film is attributed to the hydrocarbon overlayer caused by the absorption from an environment, which is generally found in XPS analysis of a sample exposed to the ambient air. The high-resolution XPS spectra of Ni 2p core level spectrum consists of satellite and two sublevel peaks of Ni 2p3/2 and Ni 2p1/2 occurred by spin–orbit splitting. The sublevel peaks are contained with six deconvoluted peaks (Ni 1-Ni 6), which was assigned to Ni$^{2+}$ and Ni$^{3+}$ [26–28]. The O 1s core level spectrum of NiO film was resolved into five peaks (O 1-O 5) as shown in figure 5(c). The higher intensity peak at the lower BE was defined to O$^{2-}$, associating with the stoichiometric of NiO [27]. On the other hand, the lower intensity peaks at the higher BE are attributed to the surface contamination [27, 29]. Figure 5(d) shows the C 1s core level spectrum consisted with four deconvoluted peaks (C 1-C 4). The C 1, C 2, C 3 and C 4 peaks were assigned to C–C, C–OH, C=O and C=O, respectively [29, 30].

The I–V characteristics of NiO deposited on ITO glass are given in figure 6(a). The results of an investigation indicate that the I–V characteristics are linear, reflecting the ohmic contact between the NiO film and the electrodes [31]. The resistivity of 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO is calculated to be

$$\frac{E}{\alpha h \nu} = \frac{A_b}{t}$$

where $A_b$ is the absorbance and $t$ is the thickness of the samples. The plot $(\alpha h \nu)^2$ versus $h \nu$ is displayed in figure 4(c), showing that the optical band gaps of ZnO, 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO are found to be 3.21 eV, 3.51 eV, 2.79 eV, 2.45 eV and 2.19 eV, respectively. The decrease in the optical band gap of the NiO is originated by the increase in crystallite size and the transition from amorphous to crystalline. This result is in agreement with absorbance and XRD. In addition, the result is also correlated to other metal oxides reported by Ramana et al and Shariffudin et al [24, 25].

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336 MΩ·cm, 312 Ω·cm, 91 Ω·cm and 50 Ω·cm, respectively. Note that the active area of NiO film is 0.45 cm². It has been observed that the resistivity of NiO films decreased with the increase in the magnetic field due to the diminishing of porosity in the NiO film as investigated by the SEM. This can be explained that the decrease in a porosity leads to the improvement of an electron diffusion, resulting in the enhancement of electrical conductance of the semiconductor [32]. To gather more information on the electrical properties of NiO films, we use Hall effect along with the van der Pauw method demonstrated in figure 6(b). The results of the measurement were shown in table 2. It was found that the values of the Hall coefficient are positive, which is the characteristic of p-type semiconductor. The increase in carrier concentration was caused by the interstitial of oxygen in NiO film [33]. Unfortunately, the electrical characteristic of 0 mT NiO films is unable to measure due to a high resistivity. Figure 6(c) illustrates the I–V characteristics of the NiO film on ZnO/ITO substrates.
measured in the range of ±2.5 V at room temperature. The non-linear curves of the devices verify the achievement of the p-n junction. In addition, 250 mT NiO/ZnO produces the highest forward current. This behavior is related to the low defects in the quasi-neutral region, which is correlated to the dislocation density as calculated from XRD [11].

Figure 6. (a) I-V characteristic curves of 0 mT NiO, 125 mT NiO, 250 mT NiO and 375 mT NiO films. (b) Schematic diagram of the van der Pauw method for Hall effect measurement. (c) I–V characteristic curves of NiO/ZnO heterojunction. (d) Semi-log plots of I–V characteristic of NiO/ZnO heterojunction.

Table 2. Electrical properties of NiO films by sparking method under a magnetic field.

| Sample name | Resistivity (Ω.cm) | Hall coefficient (cm²/C) | Carrier concentration (cm⁻³) | Hole mobility (cm²/V.s) |
|-------------|--------------------|--------------------------|-----------------|-----------------|
| 0 mT NiO    | 336 × 10⁶          | —                        | —                | —               |
| 125 mT NiO  | 312                | 41.4                     | 1.51 × 10¹⁷      | 0.133           |
| 250 mT NiO  | 91                 | 26                       | 2.4 × 10¹⁷       | 0.286           |
| 375 mT NiO  | 50                 | 24.2                     | 2.58 × 10¹⁷      | 0.484           |

Table 3 shows different deposition methods of NiO films and properties of the NiO/ZnO heterojunctions. The rectification ratio of 250 mT NiO/ZnO heterojunction was almost equal to the rectification ratio of NiO/
The NiO/ZnO heterojunction film was successfully deposited by the sparking method under a magnetic field. The application of the magnetic field in the sparking method has aligned the particles of the NiO film more uniform and tighter, resulting in the improvement of the morphology and crystal structure. Meanwhile, the optical band gap of NiO film can be controlled by varying the magnitude of a magnetic field. Furthermore, the increase in a magnetic field helps to enhance the electrical properties of the NiO films. For heterojunction, 250 mT NiO/ZnO gave better performance among all, which is supported by I-V measurements. Therefore, the non-complex system and eco-friendly for deposition of the NiO film was successful.

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