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Effect of annealing temperature on the characteristics of the modified spray deposited Li-doped NiO films and their applications in transparent heterojunction diode

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Modified spray method (m-SM) was used to fabricate 8 at%-lithium-doped NiO (NiO:8-Li) films using the nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$] and lithium nitrate ($LiNO_3$) solutions. The NiO:8-Li solutions were sprayed on glass substrates and annealed at various temperatures. It found that the resistivity was decreased and the optical bandgap value were increased as annealing temperature of the NiO:8-Li films was increased from 400 °C to 600 °C. As the annealing temperature increased, the ratio (fitting area) of Ni^{3+}/Ni^{2+} in the NiO:8-Li films decreased, which was caused by the increasing in carrier concentration. When the NiO:8-Li films was deposited on ITO glass substrates, the rectifying current–voltage (*I–V*) properties confirmed that a p–n heterojunction diode characteristic was successfully formed in a NiO:8-Li/ITO structure. The NiO:8-Li/ITO heterojunction parameters such as ideality factor (n), barrier height (φ_b), and series resistance (R_s) were determined using conventional forward bias *I–V* characteristics, Cheung's and Norde's methods. The ideality factor of 3.3, barrier height of ~0.72 eV, and the series resistance of ~0.21 kΩ were estimated using *I–V* characteristics.

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1. Introduction

Transparent conducting oxides (TCOs) have been extensively studied in recent years since they not only exhibit high optical transparency in the visible region but also have the high electrical conductivity. At present, TCOs, such as tin oxide (SnO_2), indium tin oxide (ITO), and zinc oxide (ZnO), are routinely used as transparent electrodes and window coatings for optoelectronic devices [1–3]. In contrast to n-type TCOs (like SnO_2, ITO, and ZnO), nickel oxide (NiO) shows p-type semiconductivity and has attracted much attention due to its excellent chemical stability and unique optical, electrical, and magnetic properties. NiO films have a band gap ranging from 3.6 eV to 4.0 eV and they are transparent to ultraviolet, visible, and near infrared radiation [4]. The resistivity of NiO films can be decreased by doping with monovalent impurities, such as copper (Cu), lithium (Li) and so on [5,6]. For that, NiO films have found important applications in electrochromic devices [7], organic light emitting diodes [8], gas sensors [9], dye sensitized solar cells [10], and p–n heterojunction junctions [11].

According to the literatures, NiO films can be prepared by sputtering [12], sol–gel, [13] and spray pyrolysis method (SPM) [14]. SPM is a very important method to fabricate the TCO films because spray pyrolysis is a relatively simple and atmospheric pressure deposition process, and it is an inexpensive technique for large-area coating. However, the traditional spray pyrolysis method is sprayed nickel nitrate solution onto the preheated glass substrates (>300 °C). As the substrates are heated at higher temperature, the evaporation ratio of the solution on glass substrates is too swift, resulting in the formation inferior of the NiO films. In this research, a modified spray method (m-SM) was used to develop the 8 at%-Li doped NiO (NiO:8-Li) films. The structural, optical, and electrical characterizations of NiO:8-Li films were studied in detail, and we found that the 600 °C-annealed films had better optical and electrical properties. In addition, the p–n heterojunction diodes were fabricated by depositing NiO:8-Li films on ITO glass substrates. In the past, the surveyed literatures revealed that there were no detailed reports on heterojunction parameter analysis using different methods including Sato and Yasamona's [15], Cheung's [16] and Norde's [17] methods for p-type NiO and n-type ITO. Norde proposed a method for the evaluation of the series resistance (R_s) from the forward current–voltage (*I–V*) characteristics, in which an ideal diode is sought, namely, with ideality factor ($n=1$). For $n>1$, the Sato and Yasamona method used a function $F(V)$ similar to that of the

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Norde method, taking into account that n can be greater than unity. Therefore, different diode parameters such as ideality factor, barrier height, and series resistance were determined by using Cheung's and Norde's methods in this research.

2. Experimental

Corning Eagle XG glass (Corning Incorporated, NY, USA) were used as the substrates to deposit NiO:8-Li films by using the modified spray method (m-SM). Lithium-doped nickel oxide films were prepared by SM with 1 M solution. The nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$, 99.9% in purity, Alfa Aesar, America] and lithium nitrate ($LiNO_3$, 99.6% in purity, J. T. Baker, America) were mixed with deionized (D. I.) water to form the 8 at% L-NiO solutions (NiO:8-Li). The NiO:8-Li solutions were baked at 140 °C and annealed from 400 °C to 600 °C in air for 3 h for densification and crystallization. The isopropyl alcohol was added in NiO:8-Li solution to reduce the surface tension on glass or on ITO substrates. After annealing, the thickness of the NiO:8-Li films was about 350 nm as deposited on glass with an area of 20 mm × 20 mm for films' analysis and on ITO/glass with an area of 20 mm × 20 mm for hetero-junction analysis. Crystallinity of the NiO:8-Li/glass were characterized using JEOL X-ray powder diffraction (XRD) pattern with monochromatic high-intensity Cu Kα radiation ($\lambda = 0.154184$ nm). Surface morphology and roughness of the NiO:8-Li/glass were investigated by using a scanning electron microscopy (SEM) and an atomic force microscopy (AFM). The atomic bonding states of the NiO:8-Li/glass were analyzed using ULVAC-PHI 5000 X-ray photoemission spectroscopy (XPS) with the 1486.6 eV Al Kα source and the minimum energy resolution < 0.50 eV. The deconvolution of Ni $2p3/2$ and O $1s$ electron binding energy were analyzed by Gaussian curve fitting. The resistivity (ρ), hall mobility (μ), and carrier concentration (n) of the NiO:8-Li/glass were measured using the Van der Pauw method (Bio-Rad, HL5500IU) at room temperature. The optical measurements of the NiO:8-Li/glass were taken using a UV–vis spectrophotometer. The current–voltage (I–V) characteristics of the NiO:8-Li/ITO/glass heterojunction diodes were performed with a HP4156 semiconductor parameter system; and the circle aluminum (Al) electrode of 1 mm in diameter was deposited on the NiO:8-Li/ITO/glass heterojunction diodes using the e-beam evaporation.

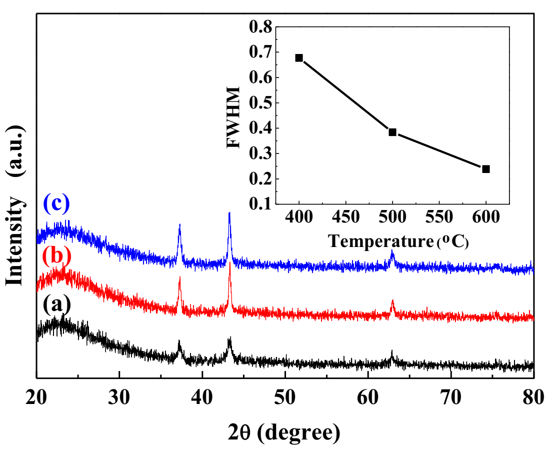

Fig. 1. XRD patterns of the NiO:8-Li films annealed at (a) 400 °C, (b) 500 °C, and (c) 600 °C.

3. Results and discussion

Fig. 1 shows the XRD patterns of the NiO:8-Li films corresponding to various annealing temperatures. Bragg peaks at $2\theta = 37.284°$, 43.184°, and 62.986° were indexed to the (111), (200), and (220) planes, respectively. In addition, diffraction peaks at (111) and (200) were indexed precisely to a bunsenite structure of NiO, which matched the JCPDS file no. 4-0835. The absence of impurity peaks revealed that the NiO:8-Li films exhibited high crystalline quality. The diffraction intensity of 600 °C-annealed NiO:8-Li films showed stronger peak intensity than those of 400 °C- and 500 °C-annealed NiO:8-Li films, indicating that the crystallization of NiO:8-Li films can be improved by increasing the annealing temperature. Inset of Fig. 1 shows the full width at half maximum (FWHM) value of the (200) peak at various annealing temperatures. The decrease of the FWHM values from 0.677 to 0.239 is attributed to the increase in the degree of crystallization caused by increasing the annealing temperature.

Fig. 2 shows the SEM images of NiO:8-Li films with various annealing temperatures. High resolution SEM revealed that 400 °C -annealed NiO:8-Li films showed a porous surface morphology, consisting of nanocrystalline grains with a randomly oriented morphology. Further increasing annealing temperature to 500 °C and 600 °C had caused the NiO:8-Li films to have large nanocrystalline grains and higher density. The average crystallite sizes were about 20 nm, 33 nm, and 48 nm as the annealing temperatures were 400 °C, 500 °C, and 600 °C, respectively. The results in Fig. 2 have also revealed an important result that the nanocrystalline grains have uniform particle sizes, and this method can be developed to grow the thin films with uniform particle sizes. By using the m-SM, the obtained surface morphology of 600 °C-annealed NiO:8-Li films had more uniform particles than those of researches by Guo et al. [18] using a sol–gel method and Juybari et al. [14] using a traditionally spray pyrolysis method and it was similar to that of the research by Jang et al. using a sputtering method [19]. Fig. 3 shows the surface roughness of the NiO:8-Li films with various annealing temperatures, measured by AFM (1 μm × 1 μm). The root-mean-square (RMS) roughness of the NiO:8-Li films increased from 2.3 to 17.7 nm when the annealing temperature was increased from 400 °C to 600 °C, and the result is agreeable to the XRD and SEM observations. As the annealing temperature is raised, the crystallinity of NiO:8-Li films are improved (Fig. 1) and the grain size becomes larger (Fig. 2).

Fig. 4 shows the transmittance spectra of NiO:8-Li films with various annealing temperatures. As NiO:8-Li films were annealed

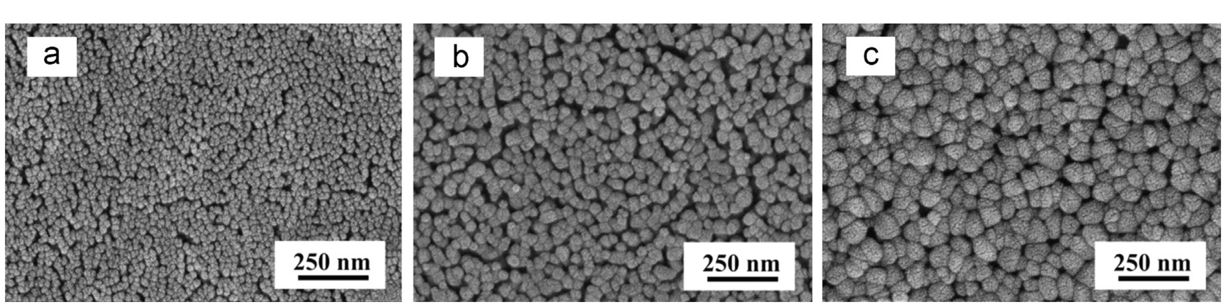

Fig. 2. Surface SEM images of the NiO:8-Li films as a function of annealing temperature: (a) 400 °C, (b) 500 °C, and (c) 600 °C.

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