IMPACT OF ANNEALING TEMPERATURES ON SURFACE MORPHOLOGY AND OPTICAL PROPERTIES OF REACTIVE SPUTTER DEPOSITED NiO FILMS

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ABSTRACT

In this work, the reactive radio frequency magnetron sputtering was used to deposit nickel oxide (NiO) thin films on Al_2O_3/glass substrate at 250 °C. Subsequently, the NiO films were subjected to annealing at different temperatures (300 °C, 350 °C, 400 °C, 450 °C, and 600 °C) in ambient air. The influence of temperature annealing on the crystal structure, surface morphology, and optical properties of NiO thin film was investigated using X-ray diffraction, scanning electron microscopy, and UV-Vis spectroscopy, respectively. The X-ray diffraction analysis reveals that the NiO films exhibit a distinct preference for crystal orientations along the (111) and (200) planes. When the temperature is raised over 450 °C during annealing, the (200) peak disappears and a new (220) peak appears. The scanning electron microscopy images clearly indicate a notable enhancement in surface roughness and a significant improvement in grain size. Furthermore, all of the samples exhibit an average transmittance of 65% across the wavelength range of 400 to 800 nm, as determined by UV-vis spectra. The optical band gap was determined to range from 3.467 to 3.658 eV. Hall measurements reveal the high film resistivity of 1.84×10^3 to 6.15×10^3 Ω.cm, which increases in accordance with annealing temperature. This outcome establishes a foundation for utilizing NiO thin films in UV-photodetectors.

ẢNH HƯỞNG CỦA NHIỆT ĐỘ Ủ ĐẾN HÌNH THÁI BỀ MẶT VÀ TÍNH CHẤT QUANG CỦA MÀNG NiO LẮNG ĐỌNG BẰNG PHÚN XẠ PHẢN ỨNG

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TỪ KHÓA

Màng mỏng niken oxit
Phún xạ phản ứng
Ảnh hưởng nhiệt độ ủ
Hướng mọc tinh thể
Tính chất quang học

TÓM TẮT

Trong nghiên cứu này, phương pháp phún xạ phản ứng được sử dụng để lắng đọng màng NiO trên Al_2O_3/lam kính ở nhiệt độ 250 °C. Sau đó, màng NiO được ủ ở các nhiệt độ khác nhau trong không khí (300 °C, 350 °C, 400 °C, 450 °C, và 600 °C). Ảnh hưởng của nhiệt độ ủ đến cấu trúc tinh thể, hình thái bề mặt và tính chất quang của màng NiO được nghiên cứu bằng nhiễu xạ tia X, kính hiển vi điện tử quét, quang phổ UV-Vis. Phân tích nhiễu xạ tia X cho thấy màng NiO phát triển theo hướng (111) và (200). Khi nhiệt độ ủ trên 450 °C, đỉnh (200) biến mất và đỉnh mới (220) xuất hiện. Hình ảnh từ kính hiển vi điện tử quét cho thấy sự tăng cường về độ nhám bề mặt và tăng kích thước hạt. Các mẫu đều thể hiện độ truyền qua trung bình trên 65% trong vùng bước sóng 400 - 800 nm, được xác định bằng quang phổ UV-Vis. Năng lượng vùng cấm quang được xác định khoảng 3,467 - 3,658 eV. Kết quả Hall cho thấy màng có điện trở suất cao từ $1,84 \times 10^3$ đến $6,15 \times 10^3$ Ω.cm, có xu hướng tăng khi tăng nhiệt độ ủ. Kết quả của nghiên cứu này tạo tiền đề cho việc ứng dụng màng NiO cho bộ tách sóng quang UV.

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1. Introduction

Nickel oxide (NiO), a famous oxide of the transitional metal group, has garnered significant interest due to its unique characteristics. NiO is an inherent p-type semiconductor with a band gap ranging from 3.4 to 4.0 eV [1]. It possesses excellent chemical stability and is more affordable compared to high-quality GaN or ZnO materials [2]. In addition, NiO characteristics as a p-type transparent conductive oxide (TCO) make it very applicable in thin film transistor devices, solar cells, and UV-photodetectors [3] – [5]. The deposited NiO thin films often present a polycrystalline structure with unpredictable growth orientations. This results in the presence of numerous defects, including grain boundaries and dislocations, which serve as traps for charge carriers. Consequently, the mobility of these carriers is greatly diminished [6]. Furthermore, it is common for the optical and electrical properties of TCO materials to exhibit a mutual nullification [7]. Hence, it is necessary to reduce the structural imperfections of NiO thin films by producing better-quality NiO thin films in order to enhance both its carrier mobility and transmittance. Therefore, the NiO thin film is regarded as a promising material for UV-photodetector applications.

Different techniques can be employed to produce high-quality NiO thin films, such as atomic layer deposition (ALD), metal-organic chemical vapor deposition (MOCVD), and RF-magnetron sputtering [8] – [10]. The RF-magnetron reactive sputtering method is commonly utilized for producing high-quality NiO films due to its cost-effectiveness, ability to easily modify parameters during deposition, and repeatability of the process. Several publications address the optimization of oxygen flow, growth temperature, substrate temperature, and annealing temperature to fabricate NiO thin films [11] – [13]. Still, there have not been many works on the study of annealing temperature in ambient air. Heating in air can effectively enhance the crystalline quality, optical characteristics, and electrical properties of NiO films, resulting in a substantial reduction in manufacturing costs throughout processing.

This study involved the deposition of NiO thin films onto Al_2O_3/glass substrates using RF-magnetron reactive sputtering. The films were then subjected to annealing at various temperatures (300 °C, 350 °C, 400 °C, 450 °C, and 600 °C). Then, the influence of heating temperatures on crystal structure, surface morphology, transmittance, and optical band gap of NiO films was investigated via different characterization techniques.

2. Experiment

The reactive RF-magnetron sputtering was used to deposit NiO thin films onto Al_2O_3/glass substrates. The glass substrates were first cleaned with acetone, ethanol, isopropyl alcohol. Next, they underwent preheating in a vacuum chamber at a temperature of 150 °C and a pressure of 10^{-5} Torr. This step helped to eliminate any remaining water vapor and contaminants. A buffering layer of Al_2O_3 of about 100 nm thickness was prepared on glass substrates from commercial ceramic target at substrate temperature of 100°C, sputtering power of 70 W, deposition time of 30 minutes and oxygen flow of 30 sccm. The NiO thin films were deposited at 250 °C in one hour. The power output and the oxygen flow rate were maintained at 100 W and 30 sccm, respectively. Then NiO films conducted annealing at different temperatures from 300 °C to 600 °C. The X-ray diffraction (XRD) technique was used to investigate the structural characteristics of NiO films. The scanning electron microscope (SEM) was used to examine the surface morphology of the films. The optical characteristics of NiO thin films were analyzed using UV-Vis spectroscopy. The electrical properties of NiO films were studied by Hall measurements.

3. Results and discussions

Figure 1 displays the XRD patterns of NiO films after being annealed at various temperatures: 300°C, 350 °C, 400 °C, 450 °C, and 600 °C. The face-centered cubic (111) and (200) preferential peaks appeared in most samples [14]. As the heating temperature got higher, the diffraction peaks became clearer and sharper. In addition, increasing the heating temperature resulted in an enhancement in the crystalline sizes of the (111) peak from 7.462 nm to 21.149 nm. With 400 °C sample, the (200) peak became dominant over the (111) counterpart. This indicated that, at this temperature, the NiO films were preferrentially grown in the (200) direction, consistent with the previous work [13]. However, when temperature got even higher, the (200) peak decreased. Interestingly, the (200) peak vanished and the (220) peak emerged when the annealing temperature rose to 600 °C. A summary of the crystal structure data derived from XRD patterns was shown in Table 1. The XRD results presented evidently the significant influence of annealing temperature on the growth orientation and crystallinity. This could explained by the surface energy: the provision of thermal energy enhanced atom mobility, facilitating their arrangement in a favorable location for achieving high-quality crystallinity [15] – [17].

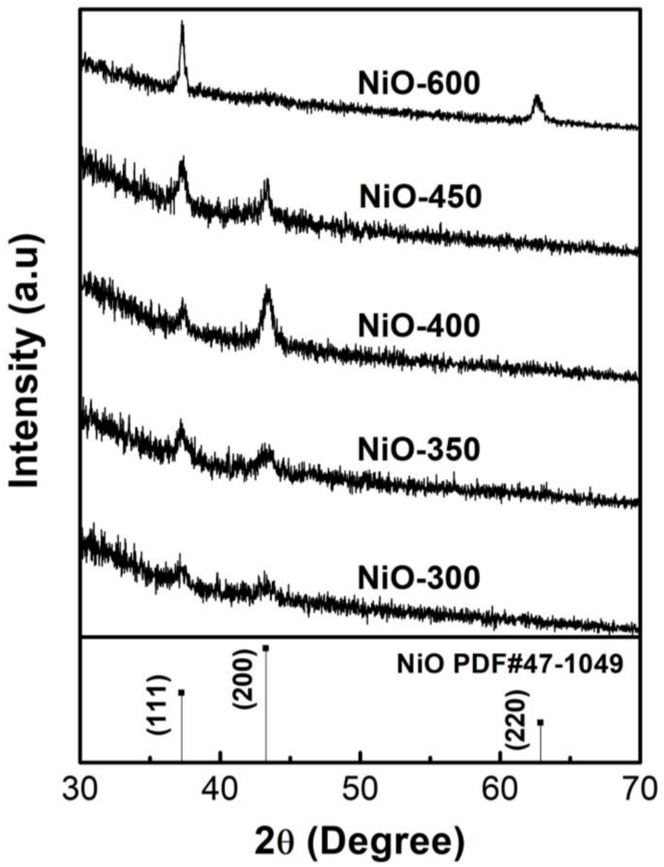

Figure 1. *XRD patterns of NiO thin films with the various annealing temperatures of 300 °C, 350 °C, 400 °C, 450 °C, and 600 °C, respectively*

Table 1. *Details from the XRD spectra about the structural attributes of the NiO (111) peak*

Samples	2θ (°)	FWHM (°)	Grain sizes (nm)	ε (10^{-3})	*d*-spacing (Å)
NiO-300	37.132	1.123	7.462	14.593	2.420
NiO-350	37.244	0.942	8.899	12.202	2.413
NiO-400	37.288	0.797	10.521	10.308	2.410
NiO-450	37.247	0.757	11.083	9.796	2.412
NiO-600	37.289	0.397	21.149	5.127	2.409

The XRD diffraction patterns provided additional information on the structural properties of the material, such as the *d*-spacing, average crystal size (*D*), and micro-strain (ε) [18], [19]. The calculated *d*-spacing values of the (111) peak were 2.420, 2.413, 2.410, 2.412, and 2.409 Å, corresponding to the annealing temperatures of 300 °C, 350 °C, 400 °C, 450 °C, and 600 °C, respectively. The values tended to decrease with increasing annealing temperature. The d-spacing of NiO-450 was compatible with the value of bulk material NiO (d = 2.412 Å) [20]. The phenomenon could be elucidated by the presence of defect density as well as micro-strain of crystal structure. The dislocation density was inversely proportional to the square of the average crystal size [18]. Therefore, raising the annealing temperature led to a considerable decrease in dislocation density, indicating a reduction in oxygen or nickel defects due to the influence of annealing temperature [16].

In addition, the micro-strain values in the NiO thin film reduced steadily from 14.593×10^{-3} to 5.127×10^{-3} as the annealing temperature increased from 300 °C to 600 °C. The presence of micro-strains arose from the stretching and compressing occurring in the crystal lattice at various annealing temperatures, as well as a variation in thermal expansion coefficients between the NiO film and the glass substrate [16], [22]. Consequently, increasing the annealing temperature leads to a significant enhancement in the crystal quality of the NiO thin film, resulting in a significant decrease in defect density and micro-strain, as shown in Table 1 [21]. Notably, the NiO film annealed at 450 °C delivered the highest crystalline quality and (111) direction crystallization intensity.

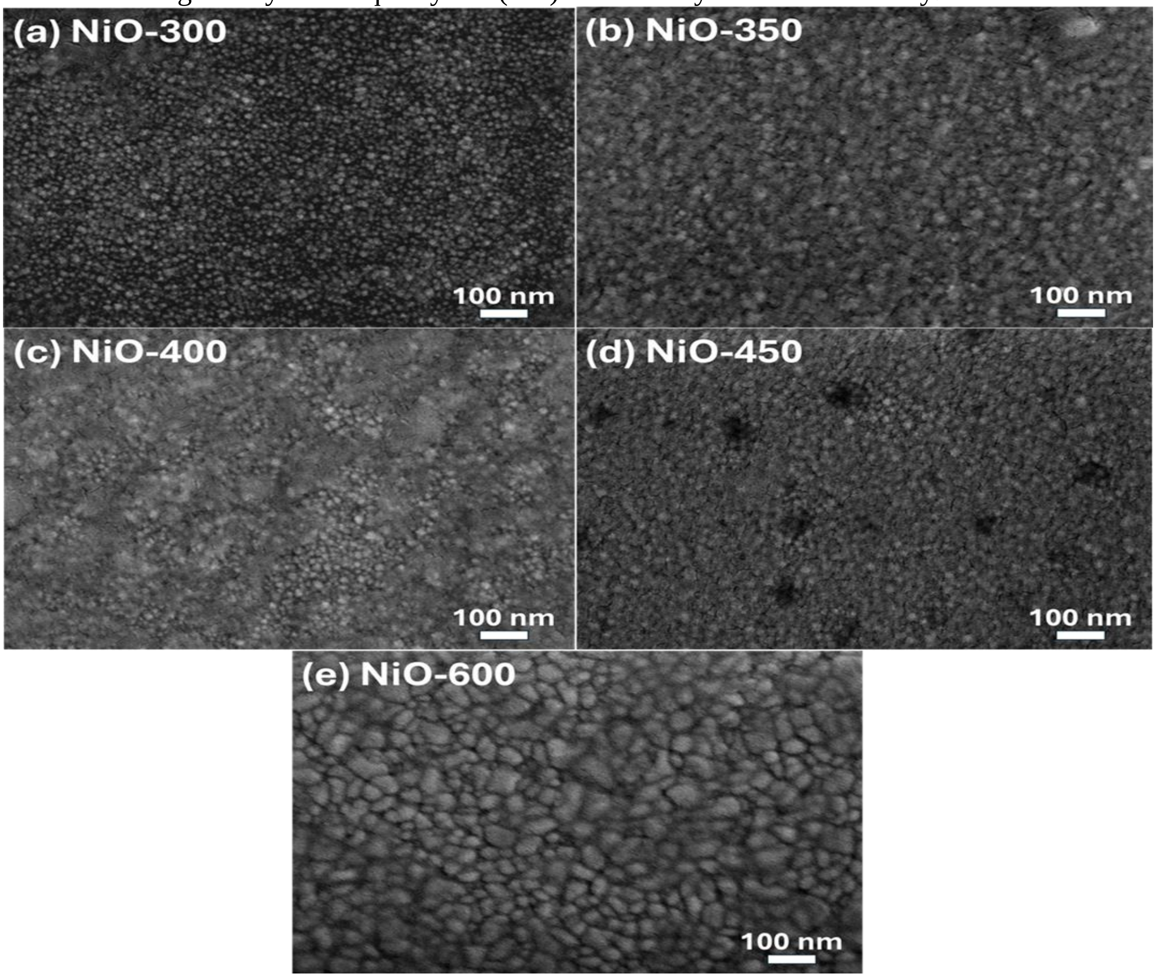

Figure 2. *SEM images of NiO samples with the annealing temperature (a) 300 °C (NiO-300), (b) 350 °C (NiO-350), (c) 400 °C (NiO-400), (d) 450 °C (NiO-450), and (e) 600 °C (NiO-600)*

The surface morphology and grain size of NiO films were significantly influenced by the annealing temperature as displayed in Figure 2. As the annealing temperature rose from 300 °C to 400 °C, the average grain size varied from 7.4 nm to 10.5 nm. Upon increasing the annealing temperature to 450 °C, the surface morphology exhibited higher roughness value and a notable crystallinity enhancement. In addition, the film grain sizes increased to 11 nm after being heated to 450 °C, whereas the particle sizes for NiO at 600 ^{o}C jumped to 21 nm. By raising the annealing temperature, the Ni and O atoms gained additional energy, allowing them to diffuse continuously on the surface of the substrate [23], [24].

The transmittance of NiO films at different annealing temperatures in the wavelength range of 300 to 1000 nm were illustrated in Figure 3a. NiO films all demonstrated high absorption in ultraviolet region and increasingly high transmission in visible and infrared parts. In the wavelength range from 400 nm to 800 nm, NiO films presented an average transmittance of over 65%. The optical transmittance of NiO films was improved due to the higher crystallite size, the improved crystallinity. Furthermore, the ability of interstitial O atoms to diffuse out helped to decrease the defect concentrations and increase the film transmittance [25] – [27].

The optical band gap values (E_g) were derived from absorption spectra using the Tauc relation and depicted in Figure 3b. The bandgap values were determined to be 3.508, 3.493, 3.467, 3.609 and 3.658 eV in compliance with annealing temperatures of 300, 350, 400, 450 and 600 oC, respectively. These values were consistent with prior works [18], [28]. The optical bandgap energy tended to increase as the annealing temperature increased. This could be associated with a decrease in the number of unsaturated bonds during the annealing process. The decrease of unsaturated bonds (such as Ni vacancies) could lead to a decrease of local state density in the energy band structures and the bandgap expansion [29].

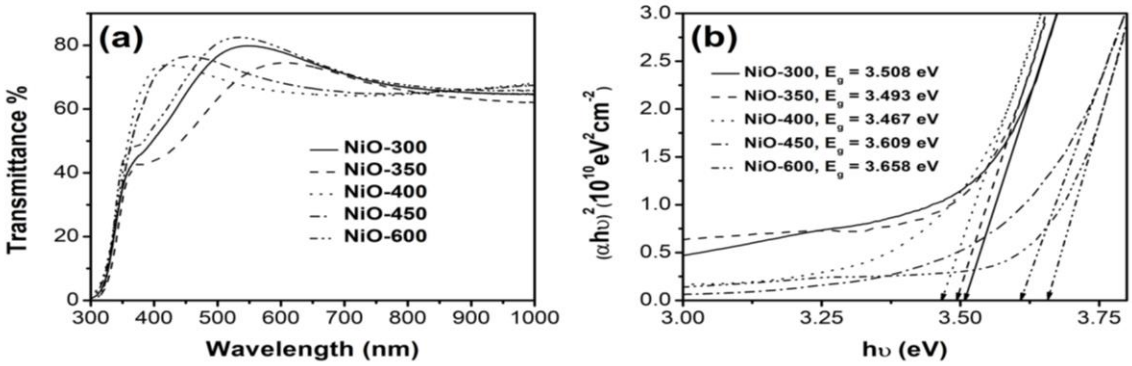

Figure 3. *NiO thin-film optical transittance spectra (a) and optical bandgap (b) at various annealing temperatures.*

The electrical properties of NiO films annealed at different temperatures were studied by Hall measurements. The impact of annealing temperature on resistivity (ρ), carrier concentration (p), carrier mobility (μ_H) was summarized in Table 2. The results demonstrated the p-type characteristic of all sputtered NiO films. The film resistivity values spreaded out from 1.84 × 10^3 to 6.15 ×10^3 Ω.cm. The resistivity tended to increase when the annealing temperature was from 300 to 450 ^{o}C and decrease at 600 ^{o}C. This revealed that when the annealing temperature was above 300 ^{o}C, a large amount of oxygen diffused out and the NiO film gradually reached the chemical equilibrium state. This decreased the concentration of Ni vacancies and instertitial O, resulting in an increase of resistivity [30]. In addition, the resistivity values of the NiO film relied on two critical parameters, namely the carrier concentration and Hall mobility. The value of the carrier concentration varied from 1.84 ×10^{12} to 6.15 ×10^{12} cm^{-3} and the Hall mobility expanded from 442 to 551 cm^{2}V^{-1}s^{-1} as the annealing temperature was from 300 to 600 ^{o}C. When the annealing temperature increased from 300 to 450 ^{o}C, the carrier concentration tended to decrease with increasing the annealing temperature due to improved crystallinity, reduced Ni and O defects within the crystal structure [31]. Besides, the carrier mobility of NiO tended to decrease due to the diffusion of oxygen and nickel atoms in NiO. This generated huge oxygen and nickel vacancies, serving as the scattering centers for the carriers [32]. Especially, at annealing temperature of 600 ^oC, both carrier concentration and mobility presented quite good values. They both contributed to the improvement of NiO film resistivity, in line with the previous report [16].

Table 2. *Electrical and optical parameters of NiO/Al$_2$O$_3$/glass samples*

Sample	Optical properties		Electrical properties			
	T % (400 – 600 nm)	E_g (eV)	Carrier type	ρ ($\times 10^3\,\Omega.\mathrm{cm}$)	μ_H ($cm^2V^{-1}s^{-1}$)	n ($\times 10^{12} cm^{-3}$)
NiO-300	71.73 %	3.508	p	1.84	483	7.05
NiO-350	74.51 %	3.493	p	1.98	457	6.92
NiO-400	69.80 %	3.467	p	3.93	472	3.37
NiO-450	73.07 %	3.609	p	6.15	442	2.30
NiO-600	75.16 %	3.658	p	2.61	551	4.35

4. Conclusion

In conclusion, the reactive RF-magnetron sputtering was employed to deposit NiO thin films onto glass substrates. The as-prepared samples were subjected to different annealing temperatures in air environment. The crystallinity, average grain sizes and roughness were greatly enhanced as the annealing temperature increased from 300 °C to 600 °C. Additionally, there was a crystal growth direction alteration in association with the increase of surface energy. The NiO films exhibited maximum transmittance of over 70% on average and decreased as the annealing temperature increased. The optical band gap energy values were measured to be approximately 3.658 eV. In addition, the resistivity of the annealed NiO film reaches a value of 1.84×10^3 to 6.15×10^3 Ω.cm, a load particle concentration of ~ 10^{12} cm^{-3} and a flexibility of 442 to 551 cm^2/V.s when the base temperature is from 300 – 600 ^oC. The results are highly significant in the utilization of NiO thin film in UV-photodetector applications.

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