Deposition of NiO Thin Films by Sol-Gel Spin Coating: Effect of Annealing Temperature on Structural, Optical and Electrical Properties

N. Parimon, M. H. Mamat, A. S. Zoolfakar, M. F. Malek, I. B. Shameem Banu, N. Vasimalai, and M. Rusop

***Abstract*—Nickel oxide (NiO) thin films were deposited by sol-gel spin coating approach and annealed at different temperatures of 300 °C, 400 °C, and 500 °C. Herein, the influence of the heat-treated process on NiO thin films to the morphological, structural, optical, and electrical properties were investigated. The field emission scanning electron microscopy reveals that the grain morphology is clearly visible with a random orientation at the highest temperature of 500 °C. Subsequently, the grains are gradually blurred and not clearly seen with the decreases of the annealing temperature. The X-ray diffraction pattern shows that a strong intensity peak of NiO is observed for the sample at an annealing temperature of 500 °C indicating the enhancement of the film crystallinity is at the highest temperature. The average crystallite sizes of NiO thin films were calculated using the Scherrer formula and yielded various sizes with a maximum value of 19 nm for 500 °C-annealed sample. The bandgap energies of the samples were estimated from the Tauc's plot to be in the values between 4.08 to 4.11 eV. The electrical properties indicate that the resistance and the resistivity of NiO thin films decrease as the annealing temperature increased. From the results, it shows the potential of these NiO thin films for the application of sensing measurement.**

***Index Terms*—NiO thin films, sol-gel spin coating, annealing temperature, structural properties, optical properties, electrical properties**

I. Introduction

In a group of p-type metal oxides, nickel oxide (NiO) is one of the most investigated semiconductors with a wide bandgap (3.6 – 4.0 eV) between the valence and conduction bands. NiO has recently gained prodigious attention because of its remarkable structural, optical, and electrical properties, as well as its good chemical stability [1]. These excellent properties enable NiO to be a promising material for use in numerous applications such as gas sensors, photovoltaic solar cells, catalysts, and electrochemical devices [2-4] due to its wide and tunable optical bandgap [5].

Many studies have been conducted and showed that NiO properties can be enhanced by applying the heat-treatment process. It involves annealing process that generally leads to the complete formation of the oxide in the deposited material. According to Cayir [2], the structural, morphological, and optical properties of NiO thin films could be developed with the process of the annealing. From the result, a dominant (111) peak of NiO showed enhancement when the annealing temperature is increased. Furthermore, the grain size increased and bandgap values decreased with the increase of the annealing temperature. Akinkuade et al. [5] reported the effects of thermal treatment on structural, optical, and electrical properties of NiO thin films. They also mentioned the crystallinity of the films will get better and crystallite size increased if the annealing temperature is increased which causes the optical bandgap of the films to decrease. The resistivity of the films from 400 to 500 °C-annealed was also seen to increase due to the increase in grain size. In another investigation, Yang et al. [3] have reported that the annealing treatment and the film thickness can influence the surface morphology and structural properties of the NiO films.

Many physical and chemical techniques have been employed in the synthesis of NiO thin films such as radio frequency magnetron sputtering [1, 3], spray pyrolysis [6], e-beam evaporation [7], pulsed laser deposition [8], and sol-gel [4, 9]. Among these techniques, the sol-gel spin-coating is a viable approach to be used as a thin film-forming medium because of its distinctive advantages. It could produce a homogeneous solution with the controlled composition and microstructure, which leads to high-quality films deposited as desired [5]. This method is rarely mentioned to discuss the effect of annealing temperature on structural, optical, and electrical properties. Besides, the NiO thin films deposited through the sol-gel spin-coating attracts the attention of many studies due to the simplistic and low-cost preparation method [6, 9]. The NiO thin films could be deposited on crystalline substrates such as silicon (Si), magnesium oxide (MgO), alumina (Al_2O_3) substrates [8], and amorphous glasses.

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Therefore, in this study, a facile and effective sol-gel spin coating method in ambient conditions was employed to produce NiO thin films on the glass substrate. The films were heated at various annealing temperatures to investigate the physical and electrical properties of the thin films. The heat-treated NiO thin films are expected to be useful in many applications, including several sensors such as humidity as evidenced by our previous work [10-12].

II. Experimental Details

NiO thin films were prepared onto glass substrates (size: 2.5 cm × 2.5 cm) using the sol-gel spin coating approach at ambient conditions. Before the coating process, the glass substrates were cleaned in a hydrochloric acid solution and rinsed with deionized (DI) water. Next, the substrates were cleaned ultrasonically using solutions of acetone, ethanol, and DI water for 15 minutes each. Thereafter, the glass substrates were dried under nitrogen (N_2) gas flow using a gas blower. In this work, 0.8 M nickel acetate (precursor) was dissolved in diethanolamine (stabilizer) and ethylene glycol monoethyl ether (solvent) to produce a precursor solution. The solution was stirred for 2 hours at room temperature and used for preparing the NiO thin films. Then, the solution was deposited directly on the top of the glass substrates using the spin coater at the deposition speed of 4000 rpm for 1 minute. After deposition, the layer was dried at 250 ℃ for 5 minutes. The deposition and drying processes were repeated five times, which finally resulted in 5 layers. Likewise, we prepared three such thin films and finally, these were annealed at different temperatures of 300 ℃, 400 ℃, and 500 ℃ for 2 hours.

The morphology structures were examined by a field emission scanning electron microscopy (FESEM, Zeiss Supra 40VP). The thickness of the sample was determined using a surface profiler (KLA-Tencor P-6). The crystallinities of the NiO thin films were investigated using X-ray diffraction (XRD, PANalytical X'Pert PRO). For optical properties, ultraviolet-visible (UV–vis) spectrophotometer (Jasco/V-670 EX) was used to determine the percentage of transmittance and value of absorbance for the deposited NiO thin films. The electrical characteristics of the thin films were analyzed using a two-probe current-voltage (I-V) measurement system (model: Advantest R6243). Prior to this measurement, silver (Ag) contacts were deposited on the top of films as the electrode by a thermal evaporation system (Ulvac VPC 1100) with the thickness of the metal contact was fixed at 60 nm.

III. Results and Discussion

A. Surface Morphology

Fig. 1 (a), (b), and (c) depicts the FESEM images of the NiO thin films that were annealed at three temperatures of 300 °C, 400 °C, and 500 °C, respectively. Overall, the surface morphology shows the homogeneous grains with high density and it can be seen that the grains are randomly oriented with uniform size on the surface. The grains gradually become prominent, precise, and separate as the annealing temperature increased. Agglomeration of the particle is observed clearly in Fig. 1 (c) for NiO film annealed at a high temperature of 500 °C. It can be observed more distinctly compared to the other samples. This result shows that the grain size and the quality of the crystals are strongly dependent on the annealing temperature [2]. The thickness of the films increases with increasing annealing temperature, which manifests itself through the surface profiling measurements. The thicknesses of the thin films were 37.5 nm, 45.7 nm, and 56.2 nm for 300 °C-, 400 °C-, and 500 °C-annealed, respectively.

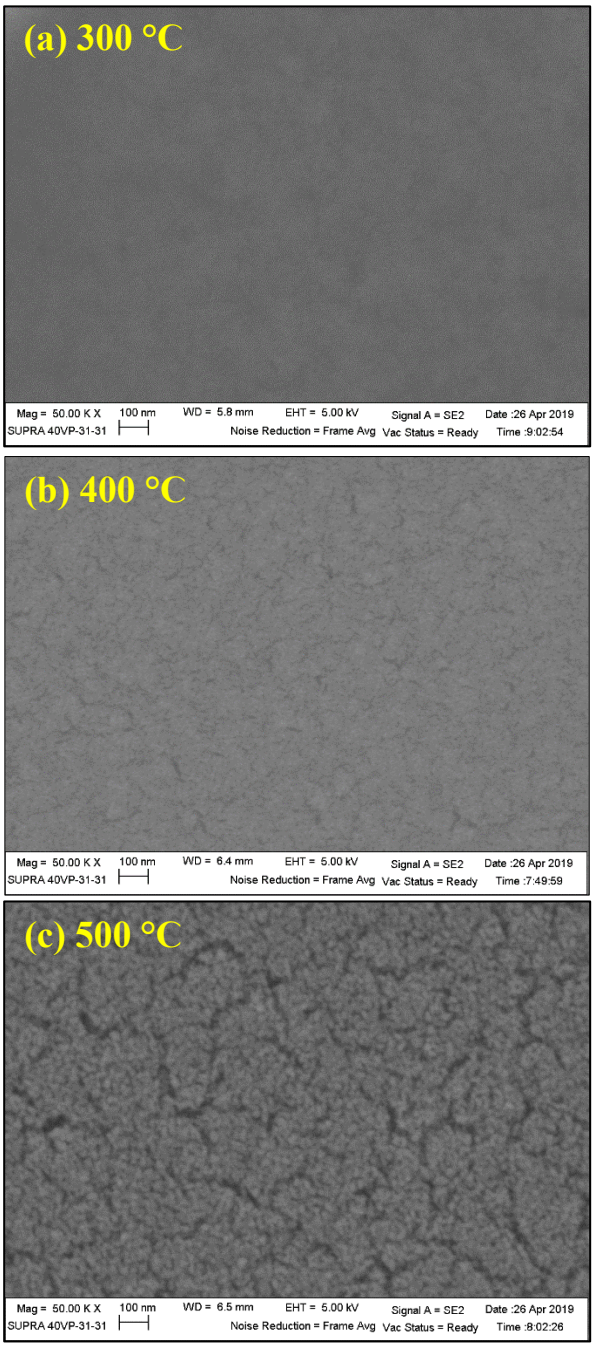

Fig. 1. Surface morphology of NiO thin films at annealing temperatures of (a) 300 °C, (b) 400 °C, and 500 °C.

B. XRD Pattern

The XRD patterns of NiO thin films at different annealing temperatures are shown in Fig. 2. The XRD peaks were recorded in the scanning range of 2θ between 20˚ to 90˚ to

verify the characteristics of the polycrystalline NiO structure. The non-annealed NiO thin film is amorphous and therefore its XRD pattern is not shown in this manuscript. This condition is in agreement with the finding by Martínez-Gil et al [13]. It can be seen from Fig. 2 that the diffraction peak appears as early as 300 ℃ of the annealing temperature but with low intensity. The intensity of the diffraction peak increases as the annealing temperature increases. The observable peak at 300 ℃ could be attributed to the 0.8 M precursor concentration used, which provides sufficient NiO molecules for lattice rearrangement of the crystallization even at a low temperature. As stated by Sahoo et al., the 0.5 M precursor of nickel acetate can be crystallized to an oxide state at annealing temperature as low as 350 ℃ [14]. In another study, the NiO samples prepared using the SILAR method at various annealing temperatures showed polycrystalline structure including those without annealing process or as-grown samples [2]. The increment of the intensity as the annealing temperature increased is consistent with the result reported by Diha et al. [4]. Further, it shows the crystallinity quality of the film improves as the annealing temperature is increased. As stated by Ghougali et al. [15], the increase in the intensity of the diffraction peaks shows that the crystallinity of NiO is good. Therefore, the best crystalline quality of the film is achieved at the highest temperature of 500 °C as its XRD pattern exhibits dominant and stronger peaks compared to other samples. Five diffraction peaks were recorded in the XRD pattern of the NiO thin film at 500 ℃, which corresponding to the cubic β-NiO (JCPDS No. 047-1049). These peaks at 36.9˚, 43.0˚, 62.6˚, 75.2˚, and 79.0˚ were indexed to the (111), (200), (220), (311), and (222) crystal planes, respectively.

The polycrystalline and cubic β-NiO can also be indexed to both 300 °C- and 400 °C-annealed films. The 300 °C- and 400 °C-annealed NiO films exhibit dominant (200) peak at approximately 42.5˚ and 43.0˚, respectively. However, the 400 °C-annealed sample shows a sharper peak than the 300 °C-annealed sample. When the annealing temperature is increased from 300 °C to 500 °C, the number of peaks is increased and the intensity of the peaks also increases. This condition indicates that the annealing temperature plays an important role in the crystallinity formation of NiO thin films.

Based on a full width at half maximum (FWHM) value, the crystallite size (D) could be calculated from the XRD data. The variation of average crystallite sizes for the annealed NiO thin films calculated using Scherer formula (1) [10] is shown in Table I.

$$D = \frac{0.94\lambda}{\beta \cos\theta} \quad (1)$$

where λ is the X-ray wavelength (1.54 Å), β is the FWHM (in rad) of the peak, and θ is the diffraction angle. The D value was acquired and calculated at (200) plane orientation as the plane is the most apparent peaks for all samples. The crystallite sizes were estimated to be 0.72 nm, 0.81 nm, and 19 nm for 300 °C-, 400 °C-, and 500 °C-annealed samples, respectively. From these values, it is clear that the average crystallite size increases as the annealing temperature is increased. This result is further reinforced from the report by Ruys et al. [15].

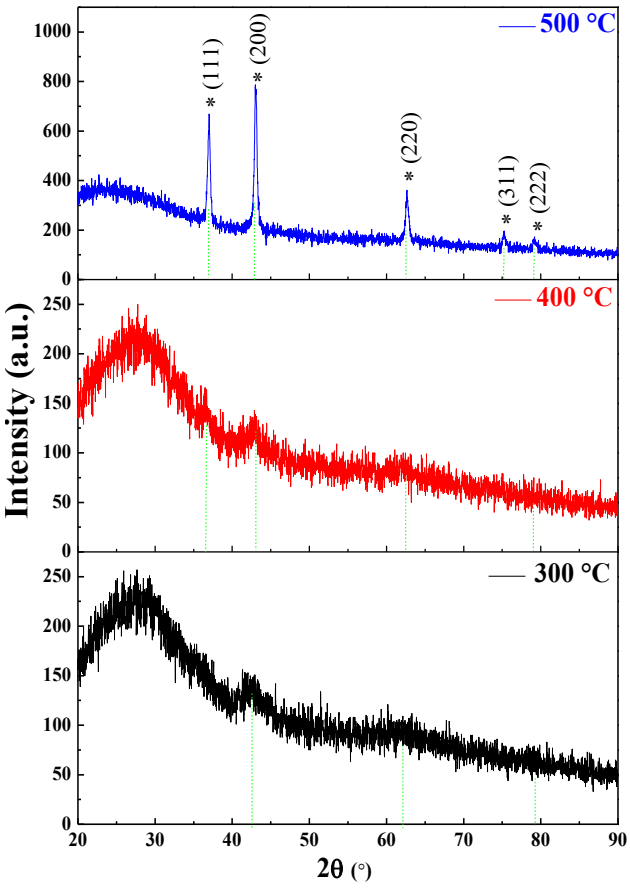

Fig. 2. The XRD patterns of NiO thin films at different annealing temperatures.

TABLE I
THE FWHM AND CRYSTALLITE SIZES OF NIO THIN FILMS AT DIFFERENT ANNEALING TEMPERATURES.

Sample (°C)	2θ (°)	FWHM, β (°)	Crystallite size, D (nm)
300	42.353	12.303	0.70
400	42.753	11.047	0.81
500	43.035	0.469	19

C. *Optical Properties*

The change in the crystallite size, which could be observed from the XRD analysis, may contribute to the changes in the optical properties [15]. As shown in Fig. 3, the transmittance spectra dependence on the wavelength ranging from 300 to 800 nm was measured. The front edge of the curves represents the intrinsic absorption of NiO [9]. It shows that the thin films exhibit high transparencies in the visible spectral region (400 – 800 nm). The average transmittance values in the visible region were estimated to be approximately 76 %, 70 %, and 83 % for 300 ℃-, 400 ℃-, and 500 ℃-annealed samples, respectively. It is observed that the transmittance percentage of NiO thin films exhibit a variation with the annealing

temperature. This variation may be due to the variation of the grain sizes when annealed at different temperatures. The highest transmittance percentage was identified at 500 °C-annealed sample, which probably due to the presence of voids between the grains that allow the light to penetrate easily.

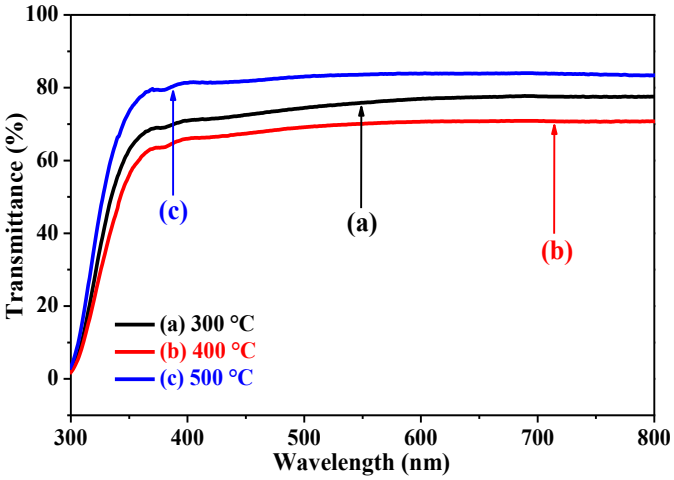

Fig. 3. Transmittance spectra of NiO thin films at different annealing temperatures.

Fig. 4 shows the absorption coefficient (α) spectra of NiO thin films annealed at various temperatures. As can be seen from all samples, the absorption values of photon energies are very low in the visible region (400 – 800 nm). However, the rapid increase in absorption occurs in the UV region at approximately 350 nm. In detail, the absorption edges of 500 °C-annealed is slightly shifted towards a shorter wavelength as compared to the samples that annealed in the lower annealing temperature. This may be due to the considerably large NiO crystalline size at an annealing temperature of 500 °. In other situations, the absorption edges of 300 °C- and 400 °C-annealed samples shifted to longer wavelengths. It can be said that the absorption coefficient is closely related to the percentage of transmission as well as the thin film thickness. This statement is substantiated by Lambert's law as expressed by the equation (2):

$$\alpha = \frac{1}{t}\ln\left(\frac{1}{T}\right) \qquad (2)$$

where α, t, and T represent absorption coefficient, thickness, and transmittance of the thin film.

Based on the calculated absorption coefficient, the Tauc's plot was constructed and presented in Fig. 5 to determine the optical bandgap (E_g). The E_g can be determined from the extrapolated linear line of the graph by plotting $(\alpha h\nu)^2$ versus photon energy ($h\nu$). The absorption coefficient *(α)*, bandgap (E_g), and incident photon energy ($h\nu$) are related by the expression (3):

$$(\alpha h\nu)^2 = B(h\nu - E_g) \qquad (3)$$

where B represents a constant. The E_g values were estimated to be 4.08, 4.09, and 4.11 eV for the 300 °C-, 400 °C-, and 500 °C-annealed samples, respectively.

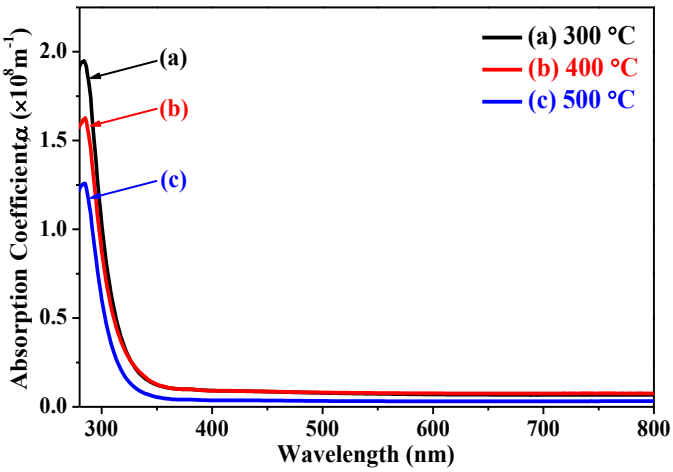

Fig. 4. The absorption coefficient of NiO thin films at different annealing temperatures.

It shows that the annealing temperature strongly affects the bandgap energies, as mentioned by Cayir [2]. From the result, the bandgap values slightly deviate from the reported values in literature, which were ranged between 3.6 to 4.0 eV. However, there a report explained the NiO bandgap was between 2.8 to 4.2 eV [16]. The difference in current E_g may be due to the very thin film thickness below 60 nm for all samples. Besides, the increase in bandgap values of the NiO films when the annealing temperature is increased from 300 °C to 500 °C may be due to changes in the crystal structure [6]. The summary of the optical properties of NiO thin films annealed at different temperatures is shown in Table II. The variation in optical bandgap showed the impact of annealing temperature on the optical properties of the NiO films.

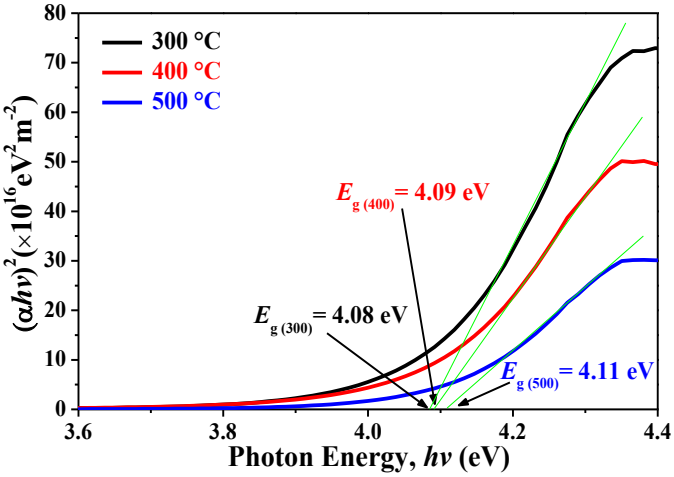

Fig. 5. Energy bandgap of NiO thin films estimated by Tauc's plot.

TABLE II
THE TRANSMITTANCE AND OPTICAL BANDGAP OF NiO THIN FILMS AT DIFFERENT ANNEALING TEMPERATURES.

Sample (°C)	Transmittance, (%)	Bandgap, E_g (eV)
300	76	4.08
400	70	4.09
500	83	4.11

D. Electrical Properties

The current-voltage (I-V) measurement result using the Ag electrode in the voltage range between −10 to 10 V at room temperature is depicted in Fig. 6. It can be seen from the I-V plots, with the increase of supply voltage, the linear current is increased and exhibit ohmic behaviour. The values of resistance (R), resistivity (ρ), and conductivity (σ) of the NiO thin films were calculated and summarized in Table III. It shows that the resistance value decreases as the annealing temperature is increased. The lowest resistance value is recorded for a 500 °C-annealed sample with a value of 5.02 MΩ. The resistivity also decreases when the annealing temperature is increased. The resistivity values obtained in this study ranges between 4.52×10^{-1} to 4.03×10^{-1} Ω·cm. Besides, all NiO thin films have good conductivity at various annealing temperatures. The highest conductivity of 2.48 $S.cm^{-1}$ is recorded at 500 °C-annealed sample. The resistivity of NiO thin film was calculated from the gradient of the I-V measurement plot using the equation (4), while the conductivity is the inverse value of the resistivity.

$$\rho = \left(\frac{V}{I}\right)\frac{A}{t} \tag{4}$$

where V, I, A, and t symbolize the voltage, current, surface area, and thickness, respectively. From the results, the resistance is found to be highly dependent on the annealing temperature, while the resistivity is dependent on the resistance, thickness of the thin film, and the surface area of the Ag electrode. According to Akinkuade et al. [5], the reduction in resistivity when the annealing temperature is increased is due to an increase in the grain size. This will lead to the reduction of grain boundaries and thus enhances carrier mobility. It is related to conductivity which represents the ability of the material to conduct electric current.

As reported by Ghougali et al. [15], the high electrical conductivity of the sample can be attributed to the high carrier concentration of the material. In addition, the good conductivity of the material also influences the good carrier mobility in the crystal structure. When the crystal structure of the film is near perfection, it will result in a reduced incidence of structural defects such as dislocations and grain boundaries. Increasing the quality of the crystals reduces the carrier scattering from structural defects, leading to higher mobility. In conclusion, the improved crystal quality reduces the carrier scattering from structural defects, thus contributing to higher mobility. From this explanation, it was in line with the results obtained in the XRD data for 500 °C-annealed NiO films, where the better crystallinity is shown for this sample can be compared to the lower temperature-annealed samples.

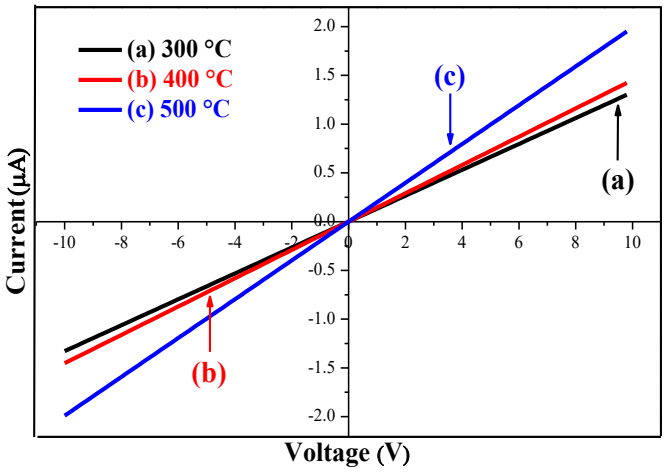

Fig. 6. I-V responses of NiO thin films at different annealing temperatures.

TABLE III
THE RESISTANCE, RESISTIVITY, AND CONDUCTIVITY VALUES OF NiO THIN FILMS

Sample (°C)	Resistance, R (MΩ)	Resistivity, ρ $\times 10^{-1}$ (Ω·cm)	Conductivity, σ ($S.cm^{-1}$)
300	7.53	4.52	2.20
400	6.89	4.49	2.23
500	5.02	4.03	2.48

IV. CONCLUSION

A facile sol-gel spin coating method has been successfully employed to deposit NiO thin films on glass substrates at different annealing temperatures. From this study, it can be suggested that the 500 °C-annealed thin film shows the best performance in terms of morphological, optical and electrical characteristics. The surface morphology analysis showed the growth of grains at high annealing temperature is favourable as agreed by XRD analysis. The XRD peak intensity of the deposited NiO thin films was enhanced by increasing the annealing temperature due to the improvement of the crystallinity of the films. All films have a cubic phase with a polycrystalline structure, with the most significant peak is at an annealing temperature of 500 °C. The impact of annealing temperature on NiO thin films could also be characterized in optical properties, especially in the variation of bandgap energy values at different annealing temperatures. Besides, the optical characterization shows that all NiO thin films have high optical transparency. Finally, the NiO thin film produced increment in conductivity after the annealing process at a higher temperature.

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