

Optical and Electrical Properties of NiO Thin Films Deposited by DC Sputtering Technique

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Abstract – In this study, Nickel Oxide (NiO) thin films were deposited on silicon and glass substrates with different thicknesses by parameter optimization using the direct current (DC) sputtering technique. The deposited NiO thin films were annealed for 30 min at different temperatures ranging from 30°C to 330°C. After annealing, the optical and electrical properties of NiO thin films were examined. Characterizations were mainly carried out by ultraviolet–visible (UV/Vis) Spectrometer and four-point probe (FPP) measurements. The minimum surface reflectance of 50 nm NiO film coated silicon substrate, annealed at 135°C for 30 min was 2.0% (at 550 nm) and the average surface reflectance was measured as 10.21% on the sample with 20 nm NiO thin film on the surface, annealed at 30°C. In case of FPP measurements, the minimum resistivity was measured as 0.01 Ωm when the thickness of NiO was 10 nm and annealed at 30°C. The maximum resistivity was observed as 4.19 Ωm on the 70 nm of NiO coated glass and annealed at 330°C for 30 min.

Keywords – Nickel oxide, Thin Film, Direct current sputtering, Electrical properties, Optical properties

I. INTRODUCTION

Nickel oxide (NiO) is an important p-type semiconductor because of its outstanding features such as large capacitance and high stability in structure [1,2]. NiO is used in supercapacitor [3], UV photodetector [4] and different types of solar cells as a hole transport layer (HTL) and antireflection layer (ARL) [5-7].

NiO thin films can be formed by methods including sol-gel [7], RF sputter [6], DC sputter [8], thermal evaporation [9], metal organic chemical vapor deposition [10]. Sputter deposition is a method of physical vapor deposition that can be defined as the deposition of particles from a sprayed surface [11]. The coating process by sputtering occurs as follows; The target material surface is bombarded with high-energy gas ions in atomic size accelerated thanks to a plasma or ion gun without using thermal energy. Thus, the atoms are sputtered off the surface and these sputtered atoms accumulate on the substrate after they turn into vapor phase [12]. A pure gas is required for this process. Argon (Ar) is generally used in this method because Ar is both noble and cost-effective also the pressure can be set in very low values, thus the desired vacuum environment can be created. In DC sputtering method, DC power supply is connected to the system. When voltage is applied to this power supply, current occurs between the electrodes. This DC generates the glow discharge, and the coating process is carried out [11]. The properties of the film in the depositions formed with this technique depend on parameters such as

deposition rate, deposition power, deposition distance, gas flow, working pressure and substrate temperature [1]. In this study, NiO thin films were formed using the DC Sputter deposition technique and optical and electrical properties of such films were investigated.

Table 1. Sputtering parameters

RF / DC	Power (W)	Distance (cm)	Substrate Temperature	Substrate	Gas Ratio (%)	Working Pressure (mTorr)	Deposition Rate (nm/s)	Thickness (nm)
DC	200	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.130	5
DC	210	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.123	10
DC	205	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.126	20
DC	200	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.130	30
DC	200	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.120	50
DC	200	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.130	70
DC	200	20	RT	Si, Glass	Ar/O ₂ : 70/30	50	0.114	100

II. MATERIALS AND METHOD

Silicon and glass were used as the coating substrates for deposition. Substrates were cleaned with an ultrasonic bath using ethanol for 5 min and dried with air. Then, the dried substrates were cleaned further with pure water for 5 min and were dried again with air. The deposition process with the DC Sputter technique was carried out by Nanovak NVTS-400 device. All deposition parameters were fixed including the substrate distance of 20 cm, gas ratio at Ar/O (70%/30%) and working pressure at 50 mTorr. NiO thin films with various thickness were deposited on the substrates: 5 nm, 10 nm, 20 nm, 50 nm, 70 nm and 100 nm, as summarized in Table 1. The deposition rate was tried to be as constant as possible for all experiments as it is very delicate. The deposition rate is 0.13 nm/s, 0.123 nm/s, 0.126 nm/s, 0.13 nm/s, 0.12 nm/s, 0.13 nm/s, 0.114 nm/s for thicknesses of 5 nm, 10 nm, 20 nm, 50 nm, 70 nm and 100 nm, respectively. After deposition, the annealing process was carried out. NiO thin films were annealed at 30°C, 135°C, 230°C and 330°C for 30 min. Annealing parameters were summarized in Table 2.

Table 2. Annealing parameters

Thickness (nm)	Temperature (°C)	Time (min)	Ambient
Variable (5-100)	Variable (30- 330)	30	Air

After annealing, the optical and electrical properties of NiO thin films were examined. Reflectance analyses were carried out on the NiO deposited silicon substrates where the electrical properties were determined on the NiO deposited glass substrates. Optical properties were measured using UV/Vis Spectrometer and electrical properties such as resistivity, sheet resistance and conductivity were measured using four-point probe (FPP).

III. RESULTS

The characteristic properties of NiO thin films deposited at different thicknesses and annealed at different temperatures were examined. The resistivity change depending on the thickness obtained using FPP is shown in Fig. 1.

The minimum resistivity value was observed as 0.01 Ωm on the glass sample with 10 nm NiO thin film on the surface and annealed at 30°C. The maximum resistivity value was observed as 4.19 Ωm on the sample coated with 70 nm NiO film annealed at 330°C for 30 min. According to the results obtained, it was observed that the resistivity value increased as the annealing temperature increases.

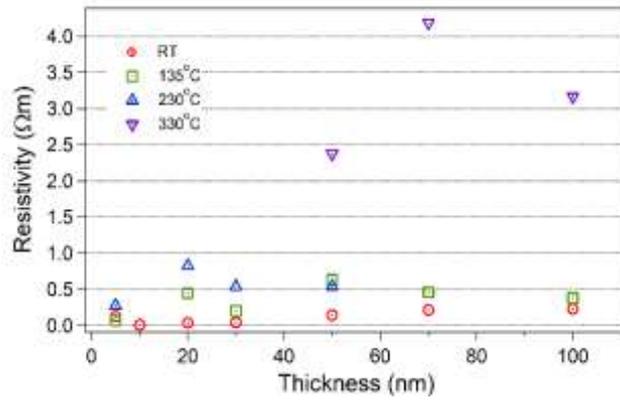


Figure 1. Resistivity change depending on NiO thickness

The maximum conductivity value was observed as 87.59 S/m on the sample coated with 10 nm NiO and annealed at 30°C. The minimum conductivity value was observed as 0.24 S/m on those of the samples with 70 nm NiO deposition on the surface which were annealed at 330°C for 30 min. According to the results obtained, it was observed that the conductivity value decreased as the annealing temperature increases. The change in conductivity depending on the thickness obtained using FPP with the same method is shown in Fig. 2.

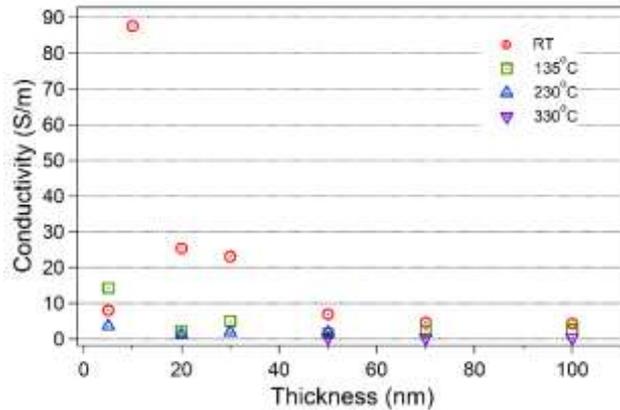


Figure 2. Conductivity change depending on thickness

The reflectance spectra values of thin films annealed at different values were determined between 380 nm and 1100 nm. Fig. 3 shows the reflectance spectra of the samples with different NiO thicknesses formed on the substrates and annealed at 30°C, where the minimum average reflectance value is obtained. The minimum average surface reflectance was measured as 10.21% on the sample that was coated with 20 nm NiO and annealed at 30°C for 30 min.

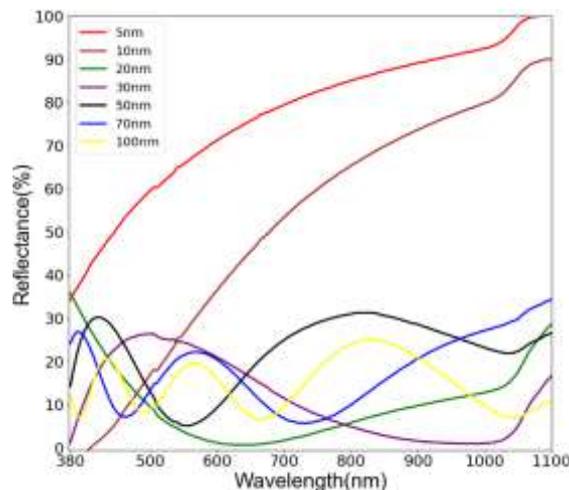


Figure 3. Reflectance spectra of NiO coated silicon substrates with different thickness, annealed at 30°C

The minimum and average reflectance values of NiO thin films deposited at 20 nm and annealed at different temperatures are given in the Table 3. The average reflectance value increases depending on the annealing temperature. Fig. 4 shows the reflectance spectra of the NiO deposited substrates with different thicknesses which were annealed at 135°C, where the minimum reflectance value is obtained.

Table 3. Reflectance values of the 20 nm NiO thin film coated silicon substrates depending on the annealing temperature

Annealing Temperature (°C)	Average Reflectance (%)	Minimum Reflectance (%)
30	10.21	0.7 (625nm)
135	11.11	0.1 (635nm)
230	12.70	1.0 (630nm)
330	13.30	1.6 (615nm)

The minimum surface reflectance was measured as 2.0% (at 550 nm) on the 50 nm NiO film coated sample, annealed at 135°C for 30 min.

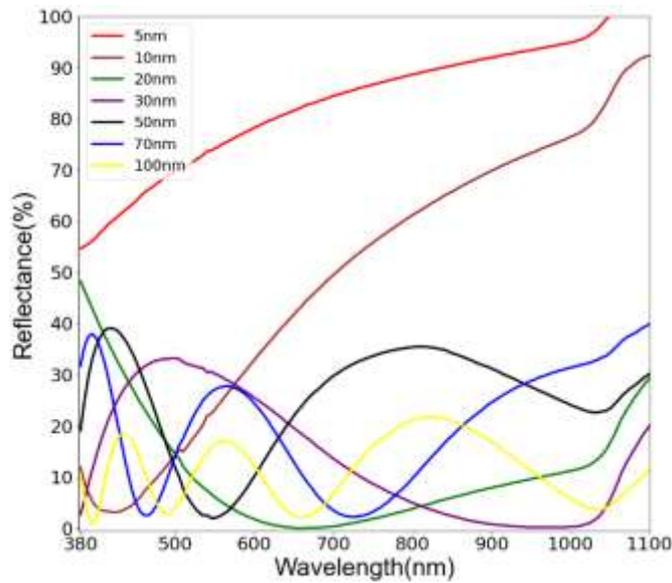


Figure 4. Reflectance spectra of NiO coated silicon substrates with different thickness, annealed at 135°C

The minimum and average reflectance values of 50 nm NiO thin film deposited surfaces and annealed at different temperatures are given in Table 4. Similarly, as on those of the samples with 20 nm NiO thin films, the average reflectance of the samples coated with 50 nm NiO increases depending on the annealing temperature.

Table 4. Reflectance values of the 50 nm NiO thin film coated silicon substrates depending on the annealing temperature

Annealing Temperature (°C)	Average Reflectance (%)	Minimum Reflectance (%)
30	22.50	5.1 (555nm)
135	25.27	2.0 (550nm)
230	26.70	1.0 (530nm)
330	28.83	0.9 (500nm)

IV. DISCUSSION

In this section, results in this work are compared with similar reported studies in literature. 70 nm NiO thin film values are taken as the basis for comparison. The resistivity of the 70 nm NiO thin films ranges from 0.2 Ωm to 4.19 Ωm depending on the increase in the annealing temperature. In the study conducted

by A. M. Reddy et al., NiO thin films were produced using DC sputter deposition where glass substrate was used in deposition process [13]. Sputter parameters were reported as: power ranging from 100 W to 200W, substrate distance of 7 cm, working pressure of 30 mTorr, temperature of 249.85°C, deposition rate between 9.25 nm/min and 21.5 nm/min. Ar and O₂ were used as sputtering gases. When the power is 200W, the measured resistivity is 0.412 Ωm [13]. In a different study conducted by Reddy et al., post-deposition annealing was applied by changing the annealing temperature between 199.85°C and 399.85°C where the NiO thin films were produced using DC sputter deposition on glass substrate [14]. Sputtering parameters were power of 150 W, working pressure of 30 mTorr, annealing temperature between 199.85°C and 399.85°C, and the resulted thickness of 350 nm. Ar and O₂ were used as sputtering gases. The measured resistivity depending on the annealing temperature is 1.358 Ωm, 0.173 Ωm and 0.315 Ωm for room temperature, 299.85°C and 399.85°C respectively [14]. In another study conducted by M. Kumar et al., NiO thin films were produced using DC sputter deposition [15]. Glass was used as the substrate with the sputtering parameters; power of 100 W, substrate distance of 15 cm, working pressure of 30 mTorr, substrate temperature between 26°C and 300°C, vacuum annealing at 300°C and duration for deposition was 1 hour. Ar and O₂ were used as sputtering gases. The measured resistivity depending on the annealing temperature is 6.2×10^{-7} Ωm and 3.6×10^{-7} for annealing at room temperature and at 300°C, respectively [15]. One can conclude that the obtained results in current study are in a good agreement with the literature.

V. CONCLUSION

NiO thin films were deposited on silicon and glass substrates with thicknesses of 5 nm, 10 nm, 20 nm, 30 nm, 50 nm, 70 nm and 100 nm. DC sputtering deposition was used in this deposition process. The deposited films were annealed at different temperatures ranging from 30°C to 330°C for 30 min. After annealing, the optical and electrical properties of NiO thin films were examined. Optical properties were measured using UV/Vis spectrometer and electrical properties were measured using FPP. As a result of these measured values, the effect of film thickness and annealing temperature on electrical and optical properties was examined. Minimum surface reflectance of 2.0% (at 550 nm) was reached on the 50 nm NiO coated silicon substrates which were annealed at 135°C for 30 min. Average surface reflectance of 10.21% was obtained on that of the sample coated with 20 nm NiO and annealed at 30°C. The minimum resistivity was determined as 0.01 Ωm on the 10 nm NiO coated glass substrates which were annealed at 30°C. Resistivity of the samples coated with 70 nm NiO was 4.19 Ωm when annealed at 330°C for 30 min. These results were in a good agreement with literature and this study can contribute to the understanding of DC sputtered NiO thin films on glass and silicon substrates.

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