

Article

Effect of Selective Lateral Chromium Doping by RF Magnetron Sputtering on the Structural, and Opto-Electrical Properties of Nickel Oxide

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Featured Application: Cr-doped NiO can be used as photovoltaic material and other optoelectronic applications as well.

Abstract: In this study, chromium (Cr)-doped nickel oxide (NiO) thin films were deposited by employing selective lateral doping of Cr in NiO by radio-frequency magnetron sputtering at different doping times ranging from 0 s (undoped) to 80 s. The structural, optical, and electrical properties of the resulting Cr-doped NiO thin films were investigated. Structural investigation from XRD patterns indicated that the grown Cr-doped NiO layer crystallized in a cubic phase. Broadening of the diffraction peak with increasing doping time from 0 s to 80 s led to a reduction in the crystallite size that varied from 23.52 nm to 14.65 nm. Compared with the undoped NiO, the diffraction peak along the (200) plane shifted from left to right as a function of doping time. This result indicated that Cr³⁺ could easily enter the NiO lattice. Results from the Hall-effect study disclosed that electrical properties of Cr-doped NiO was highly dependent on doping time. The conductivity of NiO was increased with doping time, and the highest conductivity ($8.73 \times 10^{-2} \text{ Scm}^{-1}$) was achieved at a doping time of 80 s. Finally, optical investigations revealed that as doping time increased, the optical bandgap of Cr-doped NiO films dropped from 3.43 eV to 3.28 eV. The highest Urbach energy at higher doping time indicated that crystallinity became poorer, and the degree of defects increased with increasing doping time.

Keywords: RF magnetron sputtering; selective lateral doping; NiO; thin film; doping time; microstructural defect

1. Introduction

Nickel oxide (NiO) has recently drawn great attention due to its exceptional optical and electrical properties, elevated chemical stability and high resistivity. This compound is a p-type semiconductor, naturally semi-transparent and with wide bandgap that varies from 3.6 eV to 4.0 eV [1]. Hence, modifying the structural and optoelectronic properties of NiO is crucial. Various attempts have been performed to alter the properties of transition

metal oxides. Doping of metal ions in transition metal-oxide thin films has recently been widely applied to tune the structural and optoelectronic properties [2,3]. Adopting the appropriate metal dopant that can provide enormous conducting charges will alter the properties of NiO. NiO is a transition metal-oxide semiconductor which provides a platform with Ni^{+3} and Ni^{+2} ions for metal dopants to enhance the conductivity in fabricating devices [4]. Several scientists have made attempt to enhance the key properties of NiO by using different dopants, such as copper, iron, cobalt, manganese, lithium, and aluminum [2–8]. Doping transition metal elements into NiO offers the opportunity for fine tuning of the band gap along with tailoring of the structural and electrical properties. Cr is a typical transition metal element with an abundant electron shell structure. In addition, the ionic radius of trivalent Cr^{+3} used a metal dopant (0.0615 nm) is very close to the ionic radius of Ni^{+2} (0.0690 nm) and allows the replacement of nickel ions that leads to altered NiO properties.

Several physical and chemical methods have been used to deposit NiO films. Examples of these methods include spray pyrolysis [9], radiofrequency (RF) magnetron sputtering [10,11], DC magnetron sputtering [12], chemical vapor deposition [13], electron beam evaporation [14], and pulsed laser deposition [15]. The primary benefits of RF sputtering include the production of thin films that are pinhole-free, have uniform thickness, and can be deposited over a large surface area. In this process, the properties of thin films primarily depend on the substrate temperature, sputtering pressure, RF power, system geometry, and the distance between substrate and target [16–19]. Most previous studies had focused on the doping of metal on NiO by sol-gel or chemical precipitation methods but reports on the deposition of chromium (Cr)-doped NiO by RF magnetron sputtering are rare. The purpose of this study is to create a compact, pinhole-free, and homogenous Cr doped NiO thin film using RF magnetron sputtering compared with others sol-gel or chemical precipitation methods. The concentration of Cr in the NiO was varied to investigate the optimum concentration for better film quality. This study is performed to investigate the structural, optical, and electrical properties of selective lateral doped Cr in NiO thin film by RF magnetron sputtering. For the first time, this approach (selective lateral doping) is employed to evaluate the optical, structural, and electrical properties of selective lateral Cr doped NiO thin film.

2. Materials and Methods

In RF magnetron sputtering, well-cleaned indium tin oxide (ITO) glass substrates were used to deposit Cr-doped NiO thin films. The substrates were cleaned sequentially in an ultrasonic bath by using soapy water, deionized water, methanol, isopropanol and deionized water. Then, a N_2 gas flow was applied to dry the substrates, and then the substrates were rinsed with acetone. Finally, the substrates were UV-irradiated to remove any organic residues. Selective lateral doping of Cr-NiO films was performed using a physical vapor deposition technique under a high-vacuum atmosphere through a co-sputtering technique. ITO substrates with dimensions of $3\text{ cm} \times 3\text{ cm} \times 1.5\text{ mm}$ were used for film deposition. A NiO single sputtering target and a Cr metal target were used as source materials. Preliminary purging of the sputtering chamber, followed by pre-sputtering, was performed at 50 W for 15 min to eliminate unwanted materials from the chamber. The internal pressure of the chamber was reduced to 5×10^{-6} Torr using a turbomolecular pump, and the working pressure inside the chamber was maintained at 18 mTorr throughout deposition by flowing pure argon (99.99 percent) working gas into the chamber. During deposition, the temperature of the substrate was fixed at 100°C . The RF guns were set to 50 W and 20 W for NiO and Cr, respectively. All samples were kept inside after deposition and then annealed for 3 h at the deposited temperature.

The deposition time was fixed at 90 min for the NiO target with the variation in Cr doping from 20 s to 80 s. Every 20 min interval of deposition time, we have performed the doping at one-fourth of the respective doping time. Thickness was monitored and maintained within a range 97 nm to 102 nm. The other parameters were the same for both

targets. After deposition, the samples were kept inside the chamber until the substrate temperature dropped to room temperature. This processes the oxidation at the as-grown samples. The schematic and deposition parameters used to optimize the NiO thin film are provided in Table 1, and Figure 1, respectively. The structural and crystallographic orientation of the deposited NiO thin films were measured by using the an XS-D8 Advance Cu-K α diffractometer (Bruker, Bremen, Germany). X-ray diffraction patterns were recorded at the diffraction angle (2θ) range of 20° – 70° by using the Cu K α radiation wavelength λ of 1.5406 Å. A Lambda 950 UV/Vis/NIR spectrophotometer (Perkin Elmer, Massachusetts, United States) was used to measure absorbance and optical transmission in the wavelength range of 300–1000 nm. Finally, the electrical properties of the thin film, such as carrier concentration, carrier mobility, and resistivity, were examined using Hall effect measurements (HMS ECOPIA 3000, Bridge Technology, Queen Creek, AZ, USA; probe current: 10 mA, magnetic field: 0.57 T).

Table 1. Deposition parameters (RF Sputtering) of Cr-doped NiO.

Parameters	NiO Target	Cr Target
Purity	99.99%	99.999%
Diameter of the target	50 mm	50 mm
Thickness of the target	5 mm	5 mm
Preheat temperature	100 °C	100 °C
Preheating time	90 min	90 min
Substrate temperature	100 °C	100 °C
Sputtering gas	Argon	Argon
Deposition time	90 min	20–80 s
RF power	50 W	20 W
Argon gas flow	4 sccm	4 sccm
Base pressure	5×10^{-6} Torr	5×10^{-6} Torr
Operating pressure	18 mTorr	18 mTorr
Distance between target and substrate	14 cm	14 cm

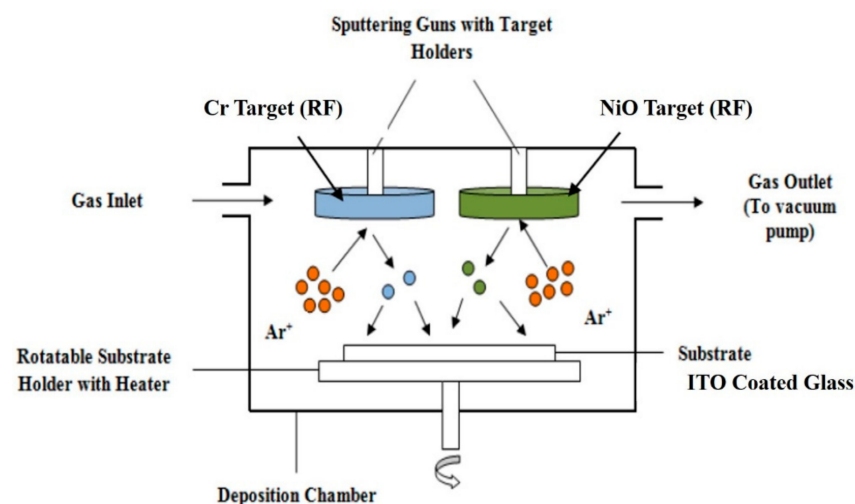


Figure 1. Schematic diagram of the sputtering chamber.

3. Results & Discussions

3.1. Structural Properties of Cr-Doped NiO Thin Films

Figure 2a shows the XRD patterns of Cr-doped NiO thin films with diffraction angles ranging from 20° to 70° at various doping times. The diffraction patterns of Cr-doped NiO films reveal two diffraction peaks at 43.3° and 62.5° , respectively, corresponding to the NiO (200) and Ni (220) orientations. These XRD patterns suitably paired the standard documented XRD spectrum in the JCPDS card No. 00-047-1049. This result indicates that the crystallization of the deposited Cr-doped NiO layer forms a cubic structure. A similar

trend has been observed in other studies in the doping of Cr in NiO [20]. Given that the doping amount is minimal, no phase corresponding to Cr is observed in the XRD pattern. The diffraction peak broadens with increasing doping time, and the amount of doping indicates that the crystallite size reduces with boosting doping time.

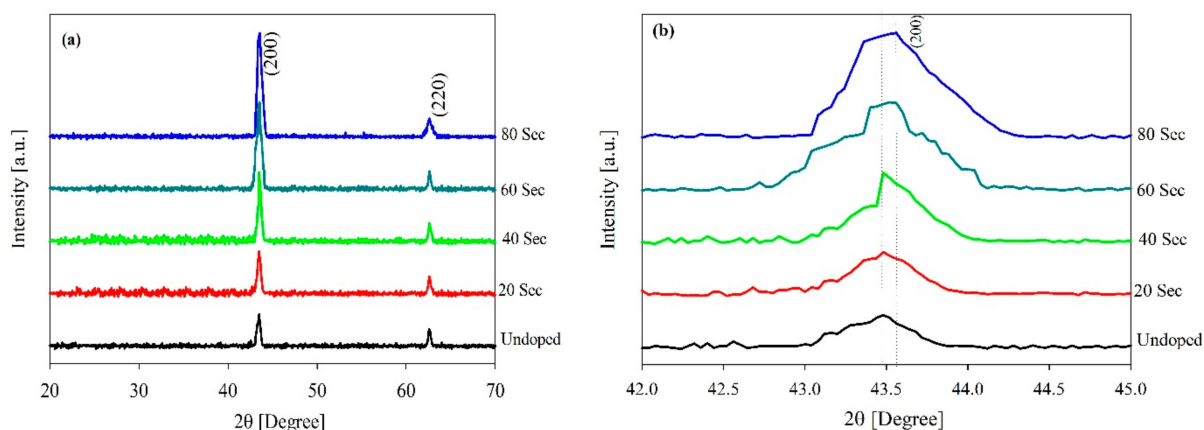


Figure 2. Structural properties of the NiO thin films at different doping times; (a) XRD pattern; (b) comparative studies on the (200) plane peak obtained from XRD pattern.

Figure 2b shows the shift of the XRD peak with the diffraction angle from 42° to 45° along the (200) plane at different doping times. Given that the radii of Ni^{+2} and Cr^{+3} at a coordination number of 6 are 0.0690, 0.0615 nm, respectively, the diffraction peaks shift to the right as the amount of Cr doping increases. The diffraction peak along (200) plane shifts to 0.08° for a doping time of 80 s, compared with the undoped NiO. This result indicates that Cr^{+3} can easily enter the NiO lattice [4].

Other structural parameters, namely, FWHM, crystallite size, microstrain, lattice constant, and dislocation density, were determined from the XRD analysis. Figure 3 shows the effect of crystallite size and FWHM of Cr-doped NiO diffraction peaks at (200) plane that corresponds to the different doping times. Crystallite size varies from 23.52 nm to 14.65 nm with the increasing doping time from 0 s to 80 s. Initially crystallite size of Cr doped NiO thin film increases with increasing doping time due to the creation of new nucleating center by the Cr atom. But at higher doping time, the crystallite size of Cr doped NiO thin film decreases and drastic change in grain size and grain boundaries. At higher doping concentration, it may be happened due to the different ionic radius of Cr^{+3} and Ni^{+2} . Average crystallite size (L) was calculated by employing Debye–Scherrer’s equation as given below [21]:

$$L_{hkl} = 0.9\lambda / (\beta \cos(2\theta)) \quad (1)$$

where, L is the crystallite size, λ is the X-ray wavelength, β is the full width at half maximum intensity of the main peak observed at 2θ (in radian), θ is the Bragg’s angle. Variations in the macrostrain and dislocation density of Cr-doped NiO thin films at different doping times were calculated using Equations (2) and (3) respectively [22–24].

$$\varepsilon = \beta / (4 \tan \theta) \quad (2)$$

$$\delta = n / L^2 \quad (3)$$

where L has the usual meaning as indicated in above and n is a factor that is very close to the unity for lowest possible dislocation density.

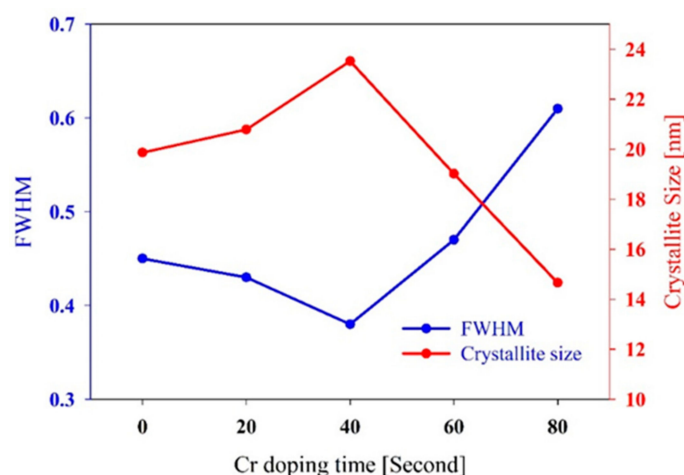


Figure 3. Effect of doping time on the FWHM and crystallite size.

The structural parameters are tabulated in Table 2. The lattice constant of the Cr–NiO thin films increased from 0.4185 nm to 0.4250 nm with increasing doping time from 0 s to 80 s. This result may be attributed to the film grains being strained at a high doping level. Increasing microstrain and dislocation density are observed at high doping times due to the decrease in crystallite size. Microstrain and dislocation density are the major factors that lead to the formation of a dislocation network in the Cr–NiO films. Strain is relaxed with the increase of dislocation density. At high doping times, increasing trend of microstrain imply poor film quality. Dislocation density also increases with deteriorating the film quality. These findings suggest that Cr doping has a significant impact on the crystal orientation and microstructure of NiO thin films. [25]. The combined effect of increased microstrain and dislocation density can be utilized to explain the stacking defect of films increases considerably with Cr doping.

Table 2. Structural parameters of Cr–NiO along the (200) plane.

Doping Time (s)	FWHM	Lattice Constant (nm)	Microstrain (ϵ , $\times 10^{-3}$)	Dislocation Density (line/cm ² [δ], $\times 10^{11}$)
Undoped	0.45	0.4185	4.924	2.539
20	0.43	0.4197	4.705	2.319
40	0.38	0.4244	4.158	1.811
60	0.47	0.4233	5.137	2.769
80	0.61	0.4250	6.661	4.664

3.2. Electrical Properties

Electrical properties of the Cr doped NiO thin films such as carrier concentration, carrier mobility, conductivity etc. at different doping times were measured through Hall measurement, and the results are tabulated in Table 3. Equations (4) and (5) explain the relationship between resistivity, carrier concentration, and carrier mobility:

$$\sigma = q\eta\mu_h \quad (4)$$

$$\sigma = \frac{1}{\rho} \quad (5)$$

where σ is the conductivity, ρ is the resistivity, q is the electrical charge, η is the carrier concentration, and μ_e is the carrier mobility. The resistivity is inversely proportional to the carrier concentration and electrical mobility.

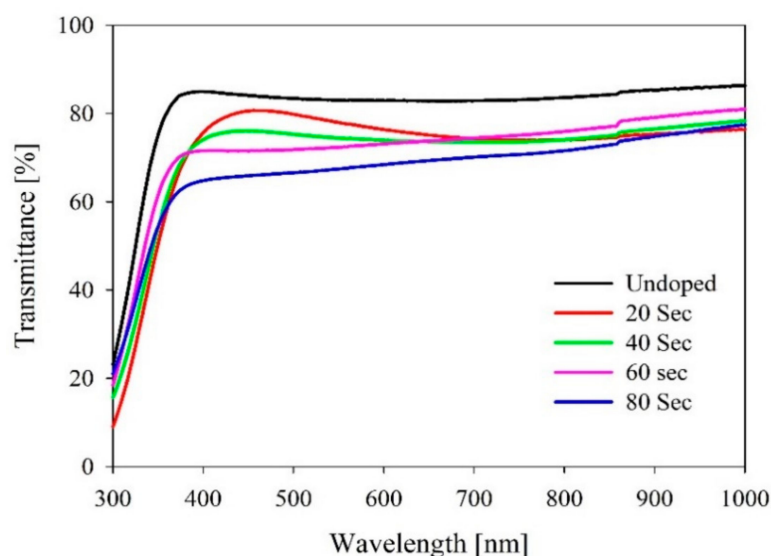
Table 3. Hall parameters of the Cr-doped NiO thin films.

Doping Time (s)	Carrier Concentration (cm^{-3})	Carrier Mobility ($\text{cm}^2/\text{V}\cdot\text{s}$)	Resistivity ($\Omega\cdot\text{cm}$)	Conductivity (Scm^{-1})
Undoped	2.85×10^{14}	10.20	2150	4.65×10^{-4}
20	6.47×10^{14}	20.90	462	2.16×10^{-3}
40	2.35×10^{15}	33.60	79.1	1.26×10^{-2}
60	5.52×10^{15}	31.6	35.9	2.79×10^{-2}
80	2.17×10^{16}	25.1	11.5	8.73×10^{-2}

The carrier concentration of the Cr-doped NiO films increases and resistivity decreases with an increase in doping time. The highest carrier concentration is observed at a doping time of 80 s. At doping time of 80 s, film resistivity became lower with moderate mobility. This result may be due to the presence of small impurities from Cr doping that decrease the bandgap of the Cr-doped NiO. Initially, the carrier mobility of the Cr-doped NiO thin films increased with doping time while the resistivity decreased. Carrier mobility gradually decreased after a doping time of 40 s, with continuously increasing doping time and decreasing resistivity. This result could be due to an improvement in dislocation density, as shown in Table 3 which increases carrier scattering and, as a result, lowers carrier mobility. Thus, electrical conductivity increases with doping time. Electrical conductivity varies from $4.65 \times 10^{-4} \text{ Scm}^{-1}$ to $8.73 \times 10^{-2} \text{ Scm}^{-1}$ when doping time is increased from 0 s to 80 s. Because of microstructural defects in the NiO crystal and surface chemical reactions, the electrical conductance of Cr-doped NiO thin films changes with doping time.

3.3. Optical Studies of Cr Doped NiO Thin Film

Figure 4 depicts the optical transmittance of Cr–NiO over a wavelength range of 300 to 1000 nm. Within the visible range, transmittance gradually decreases with increasing doping time of Cr. The optical transmittance of the undoped NiO is nearly 84.92% within the visible region but gradually decreases to ~68% as the doping time increased from 0 s to 80 s. This finding may be a result of the increase in the Cr content in the NiO thin film. Light scattering is expected to be noticeable due to the presence of numerous grain boundaries and point defects that reflect incident light.

**Figure 4.** Spectral transmittance of the Cr-doped NiO.

Optical bandgap is tabulated from the transmission spectrum of the Cr doped NiO thin films by using the well-known Tauc plot relation as follows [26]:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (6)$$

where A is the constant, E_g is the optical bandgap, α is the absorption coefficient, ν is the incident photon frequency, and h is Planck's constant.

The direct bandgap of Cr–NiO reduces from 3.43 eV to 3.28 eV with increasing doping time from 0 s (undoped) to 80 s. This change in bandgap value may be attributed to the presence of Cr or the valance defect in the NiO thin film that helps form an intraband state within the bandgap and decreases the bandgap. Narrowing of the energy band-gap in the presence of dopants is ordinary and rather detrimental but the presence of Cr dopant led to enhance the electrical properties of NiO thin film.

The exponential region between the absorption coefficient curve and the optical band edge is called Urbach tail. Because of their localized states, this tail appears in structurally disordered, poor crystalline, or amorphous materials. When plotting $\ln(\alpha)$ against the incident photon energy, the Urbach energy can be calculated by inverting the slope of the straight line, as shown in Figure 5a. The Urbach energy of the Cr–NiO films increases from 181 meV to 458 meV as the doping time increases from 0 to 80 s. At a doping time of 80 s, the highest Urbach energy is observed. This result indicates that as temperature rises, crystallinity deteriorates since the degree of defect increases. Figure 5b shows the variation in the bandgap and Urbach energy at different doping times. Urbach energy increases with doping time, but the bandgap trend is opposite to that of the Urbach energy.

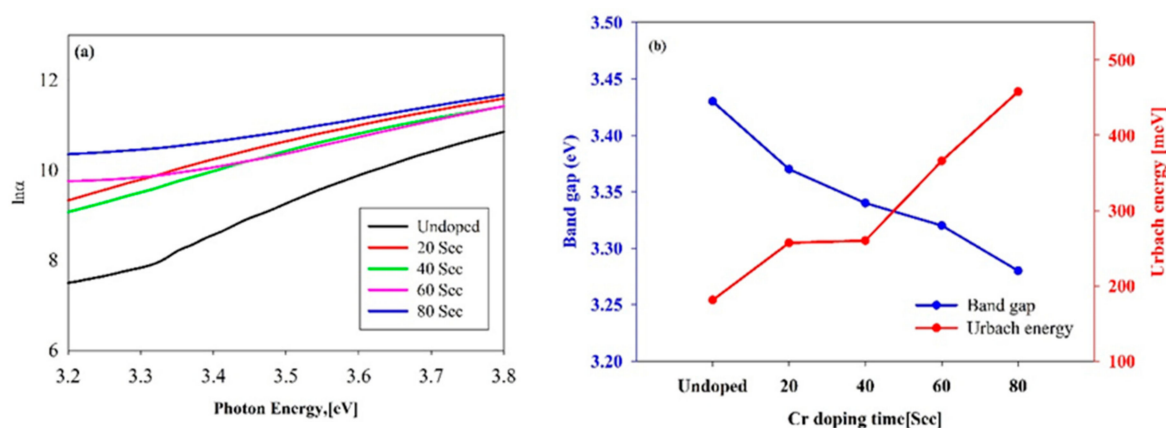


Figure 5. (a) Urbach tail and (b) bandgap versus Urbach energy of the Cr-doped NiO at different doping times.

3.4. Morphology Studies of Cr Doped NiO Thin Film

The variation of the surface morphology of the chromium–NiO thin films at different doping times were investigated through FESEM and shown in Figure 6a–e. The FESEM micrographs shows the nanoscale grain of the chromium–NiO thin films, and grain size is insignificantly dependent on doping time. FESEM micrograph clearly shows that the chromium–NiO films are uniform, cover the entire surface area. FESEM and EDS mapping confirmed that no pinholes was observed on the Cr doped NiO thin film.

EDS mapping was done to investigate the presence of chromium and pinhole. All results are presented in Figure 7a,b. Detailed EDS data have been included in Figure 7c. Though increasing the doping time improved the conductivity, reduced the optical transmittance. Both these properties are crucial for PV applications, thus as a compromise chromium-doped NiO with a doping time of 40 s at 100 °C is chosen as the optimum material for further application.

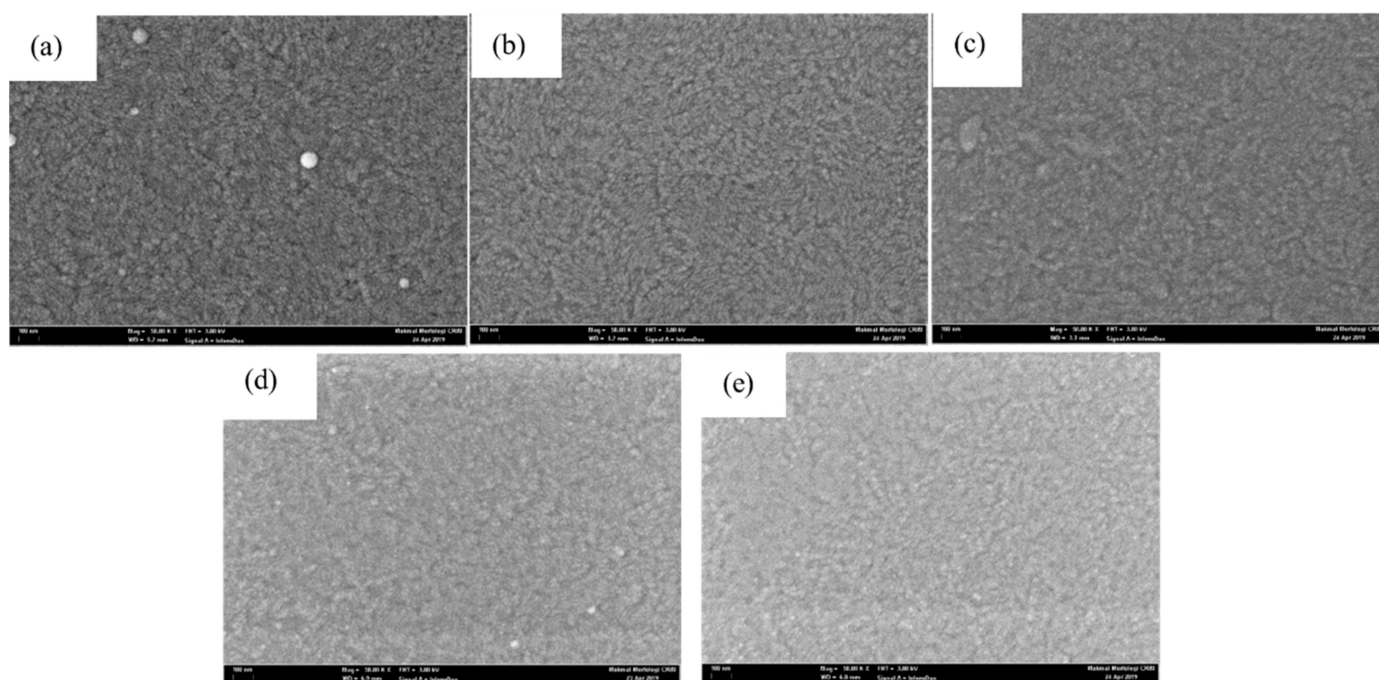


Figure 6. Surface morphology of Cr-doped NiO at different doping times (a) Undoped (b) 20 s (c) 40 s (d) 60 s (e) 80 s.

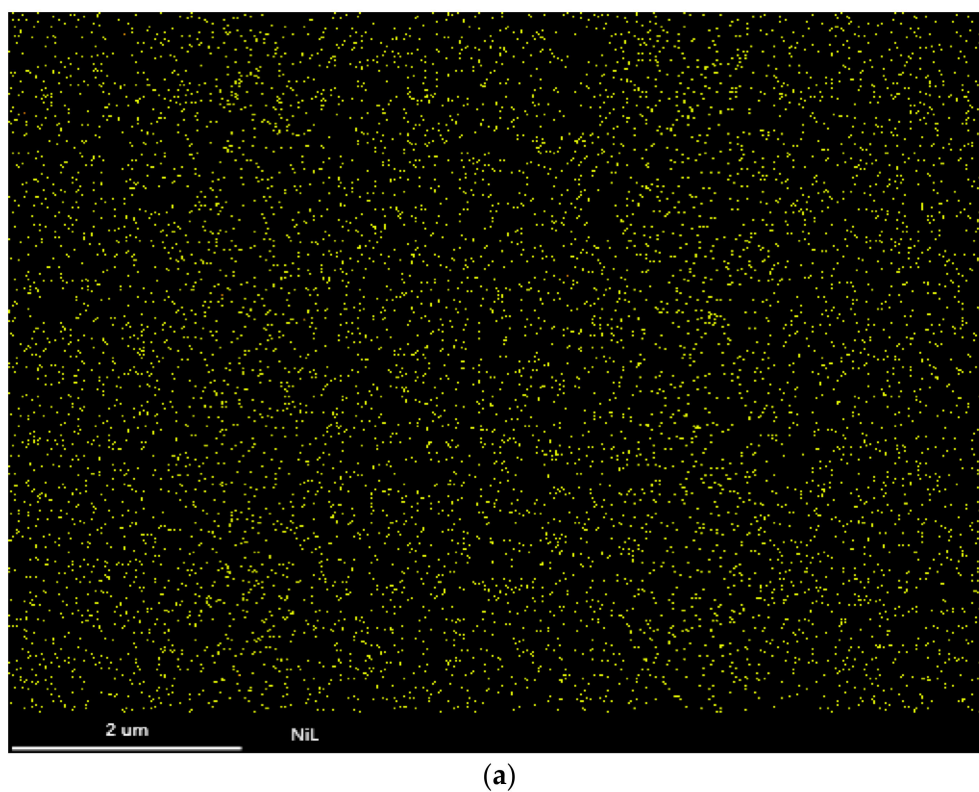


Figure 7. Cont.

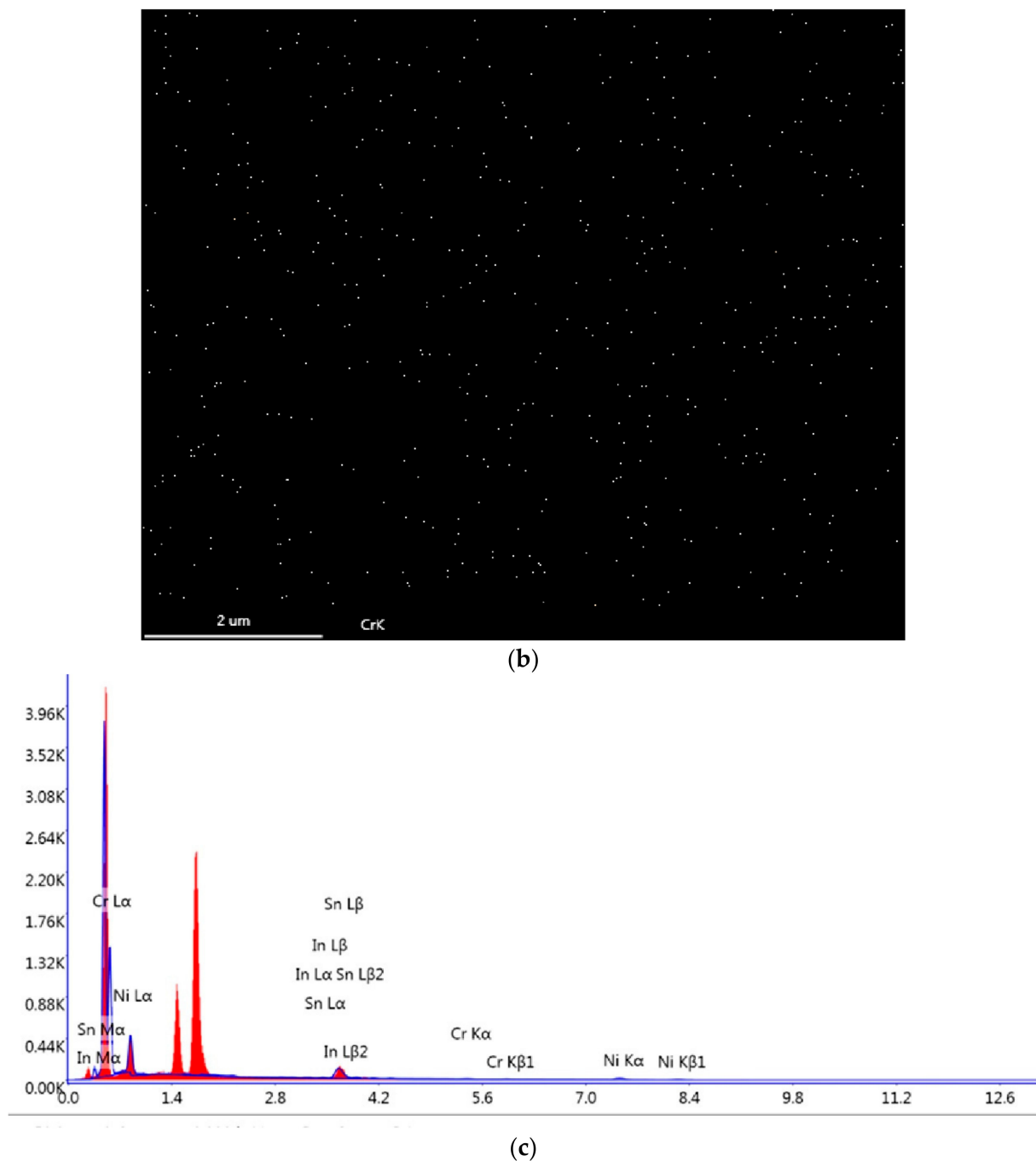


Figure 7. EDS mapping for (a) Ni (b) Cr and (c) EDS data for Cr doped NiO thin film.

4. Conclusions

The crystal quality of Cr-doped NiO significantly worsened with the increase in doping time. The lattice constant of the Cr–NiO thin films also increased from 4.185 Å to 4.250 Å as the doping time was increased from 0 s to 80 s. This finding may be attributed to the film grains being strained at high doping levels. The microstrain and dislocation density increased at high doping times due to the stacking defect of films increases considerably with Cr doping. These parameters are the leading causes of the dislocation network in the Cr–NiO films. The increasing microstrain and dislocation density indicated the poor film quality at high doping times. The conductivity of the Cr-doped NiO increased with doping time, and the highest conductivity ($8.73 \times 10^{-2} \text{ Scm}^{-1}$) and lowest resistance ($11 \text{ } \Omega \cdot \text{cm}$) were found at the doping time of 80 s. The highest carrier mobility ($33.60 \text{ cm}^2/\text{V} \cdot \text{s}$) was

observed at a doping time of 40 s. Optical studies showed that the optical transmittance of the undoped NiO was approximately 84.92% within the visible region, but gradually decreased to ~68% as the doping time was increased from 0 s to 80 s. This phenomenon may have been caused by the increase in the Cr content in the NiO thin films. When the doping time was increased from 0 s to 80 s, the direct bandgap of Cr-doped NiO was reduced from 3.43 eV to 3.28 eV, and the Urbach energy of the Cr–NiO films increased from 181 meV to 458 meV. The highest Urbach energy was observed at a doping time of 80 s. This result indicated that the crystallinity became poorer, and the degree of defect increased with doping time.

Author Contributions: M.S.J.: Writing—original draft; K.S. (Khan Sobayel): Conceptualization, formal analysis; H.M.: Writing—review & editing; T.N.: Methodology; K.A.: Writing—review & editing; H.I.A.: validation & visualization; M.S.: editing & visualization; K.S. (Kamaruzzaman Sopian): Project administration & funding acquisition; N.A.: Supervision; M.A.: Supervision & Writing—review & editing. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data will be made available by the corresponding author upon reasonable request.

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Conflicts of Interest: The authors declare no conflict of interest.

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