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Electron Beam Evaporated Nickel Oxide Thin Films for Application as a Hole Transport Layer in Photovoltaics

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Abstract: We present the growth of nickel oxide (NiO) thin films as a hole transport material in photovoltaic devices using e-beam evaporation technique. Such metal oxide layers were reactively deposited at 200 C substrate temperature using an electron beam evaporator under an oxygen atmosphere. The reactively grown oxide films through electron-beam evaporation have been optimized for carrier transport layers. Optical and structural characterizations were performed using UV–Vis spectrometry, X-ray diffraction technique, contact angle measurements, scanning electron microscopy, hall-effect measurements. The study of these films confirms that NiO layer is a suitable candidate to be used as a hole transport layer based on hall effect measurements. Morphological study using field emission scanning electron microscopy confirmed compact, uniform, and defect-free metal oxide layers growth. Contact angle measurements revealed that the films possessed semi-hydrophilic properties, contributing to improved stability by repelling water from their surfaces. The stoichiometry of the films was influenced by the oxygen pressure during deposition, which affected both their morphological and optical features. NiO films exhibited a transmittance exceeding 80% in the visible spectrum. These findings highlight the potential applications of such nickel oxide films as hole transport material layers.

Keywords: E-beam evaporation; nickel oxide; reactive growth; hole transport materials

1. Introduction

p-type metal oxide thin films are well known for microelectronic and optoelectronic devices due to their advantageous empirical properties [1-15]. NiO, in particular, is a well-established material known for its optical, electronic, and chemical stability. Several research groups have focused on optimizing the growth of NiO films for use in optical interference thin-film filters [1-3]. Numerous studies have also demonstrated the suitability of these metal oxide films for selective carrier transport, attributing this to their favorable electronic characteristics [16-25]. The development of thin metal oxide films is critical for various photovoltaic (PV) applications, where they function as essential electrical components, protective passivation layers against moisture, and optical elements, such as antireflection coatings, high-reflectance mirrors, and diverse filters. Beyond morphological aspects like uniformity and homogeneity, the refractive index of these films is a key factor influencing the spectral behavior of transmission, absorption, and reflection. Therefore, a comprehensive understanding and precise control of the properties of metal oxide films are crucial for enhancing PV device performance. Our research has demonstrated the feasibility of fabricating cost-effective nanofilms from affordable oxide materials. This innovative approach offers increased stability through strategic layer stacking, suggesting significant cost efficiency for large-scale production. In general, physical vapor deposition with low temperature developed p-type oxide materials has many advantages, including cost effectiveness and stability with a resistance to moisture. High carrier mobility with a defect free electrical interface with the absorbing layer is crucial to reduce carrier

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recombination within any device. The oxidation of nickel (Ni) in an oxygen (O_2) atmosphere is influenced by multiple factors, such as the metal's chemical characteristics and the specific conditions under which oxidation occurs. Metals exhibit diverse oxidation behaviors due to differences in their electronic structures and reactivity with oxygen. For nickel, various oxides can form, with nickel (II) oxide (NiO) being the most common under moderate temperature conditions. In contrast, nickel (III) oxide (Ni_2O_3) forms at higher temperatures and is less frequently encountered. These thin films can be synthesized using various physical vapor deposition techniques, such as sputtering [25-30], evaporation [31-35], and atomic layer deposition (ALD) [36-37]. This research introduces a method for creating metal oxide films using reactive electron beam (e-beam) evaporation of pure metals within a controlled oxygen environment. Metal oxide layers are integral to photovoltaic (PV) devices, enhancing both efficiency and stability. These layers fulfill critical roles, such as surface passivation, facilitating charge transport, and improving light absorption. For example, nickel oxide thin films are frequently utilized as hole transport layers (HTLs) or electron-blocking layers in various solar cell configurations. These materials effectively transport charge carriers while mitigating recombination at the interface between the active layer and the electrode. Additionally, metal oxide layers can be engineered to achieve optimal optical and electrical properties, including high transparency, low resistivity, and appropriate band alignment with adjacent device layers. Through careful design and optimization, these layers contribute significantly to enhancing the performance, reliability, and durability of photovoltaic systems, promoting advancements in sustainable energy technologies.

E-beam evaporation maintains vacuum integrity, which is crucial for high-efficiency devices. However, fine-tuning the growth conditions of the hole transport layer (HTL) is essential to achieve desirable optical and electrical characteristics. Perovskite materials, often used for light harvesting, are highly susceptible to moisture, which degrades the cation and anion sites, resulting in defect-prone films with poor photovoltaic performance. Thus, the development of inorganic ETL and HTL layers with hydrophobic properties is critical for encapsulating and protecting the perovskite layer, ensuring stability against water infiltration.

In this study, nickel oxide films were deposited using e-beam evaporation, with the oxygen flow rate being carefully adjusted to control the deposition pressure in the chamber. The choice of e-beam evaporation was driven by its precision in deposition rate control and its suitability for large-scale device manufacturing without interrupting the vacuum environment. This technique allows fabrication in a controlled environment to restrict performance deterioration. We have performed oxygen pressure influences on the microstructure, morphology, and optical characteristics of nickel oxide films. Optical characterization techniques, such as absorption coefficient analysis, absorbance measurements, and ellipsometry, were used to determine the bandgaps of the films. The data analysis showed a clear relationship between the surface morphology and deposition pressure. Previous research has examined the structural, optical, electrical, and micro-morphological characteristics of metal oxide thin films created through reactive electron-beam (e-beam) evaporation at different oxygen pressures, along with the effects of post-deposition annealing. A key limitation in these studies has been the need for elevated processing temperatures, which presents obstacles for producing films suitable for flexible substrates. E-beam evaporation is particularly valuable for its precise control over the deposition rate, allowing for uniform film coverage, versatility in depositing a wide range of oxide materials, and flexibility in adjusting the oxygen-to-metal ratio. In this work, we analyzed how varying oxygen pressure influences the optical properties and surface structure of the thin films produced, contributing new insights into their possible applications.

2. Materials and Methods

The main goal is to develop optimized Nickel oxide thin films (NiO) using the Denton Explorer e-beam evaporator at 1 Å/s with 100 nm under two deposition pressures, where all the films were evaporated reactively under oxygen at room temperature using e-beam evaporator. Later, samples were characterized to study the structural and morphological properties. To initiate the process, 4 inches glass substrates were cleaned in an ultrasonic bath following a degreasing protocol. This

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involved immersing the substrates sequentially in isopropanol and then rinsing with deionized water, each step lasting 5 minutes. After cleaning, the substrates were dried using nitrogen gas (N_2) flow. The deposition chamber's base pressure was reduced to 3.5 × 10^{-7} Torr using a cryo pump. During deposition, the working pressure was maintained at 2 × 10^{-4} Torr and 2 × 10^{-5} Torr by introducing a controlled flow of high-purity oxygen (99.9995%). The substrates were rotated at a speed of 5 rpm. The distance between the substrate and the source was kept constant at 100 mm. The evaporation parameters for deposition are detailed in Table 1.

Table 1. The details of the deposition condition.

Parameters	Values
Base pressure (Torr)	3.5 × 10^{-7}
Deposition pressure (Torr)	2 × 10^{-4}, 2 × 10^{-5}
Deposition rate (A/s)	1
Source material	Ni
Source material (%)	99.999
Estimated final thickness (kA)	1
Substrate heat set point (C)	200
Oxygen flow rate (sccm)	15
Substrate size	4″

3. Results

X-ray diffraction (XRD) analysis of evaporated nickel oxide thin films provides valuable insights into the crystallographic structure and phase purity of the deposited layers. Typically, the XRD patterns of NiO thin films exhibit distinct peaks corresponding to the cubic crystal structure, with prominent reflections at planes such as (111), (200), and (220).

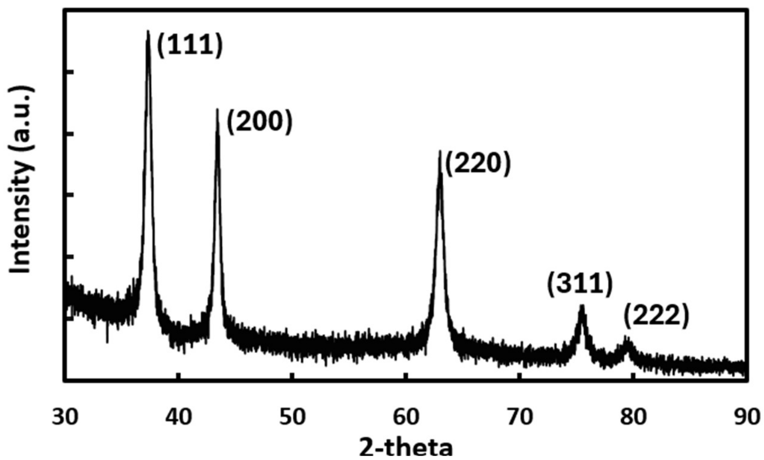

Figure 1. XRD of nickel oxide films grown at 10^{-4} Torr.

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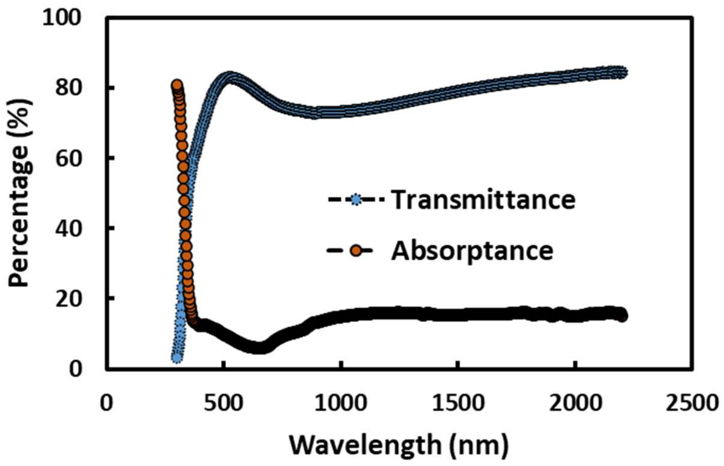

Figure 2. Optical properties of nickel oxide films at 10^{-4} Torr.

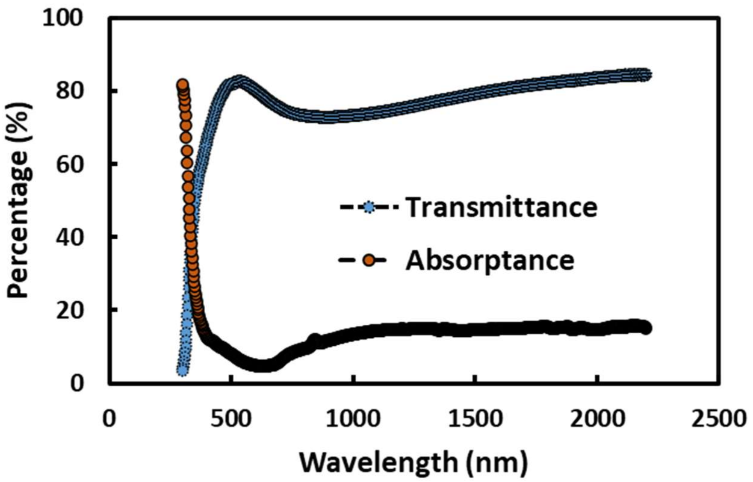

Figure 3. Optical properties of nickel oxide films at 10^{-5} Torr.

The UV-Vis spectroscopy analysis of electron beam evaporated nickel oxide (NiO) thin films reveals their optical transmittance and absorptance characteristics, which are crucial for applications in optoelectronic and photovoltaic devices. Typically, the transmittance spectra of NiO films demonstrate high transparency in the visible region, often exceeding 80%, depending on the film thickness and deposition parameters.

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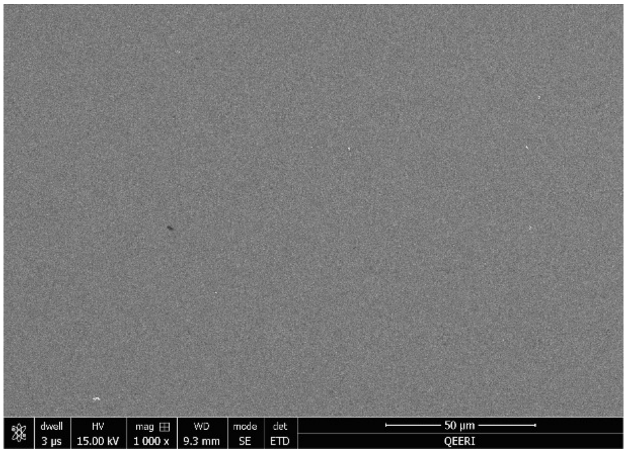

Figure 4. SEM of nickel oxide films at 10^{-4} Torr.

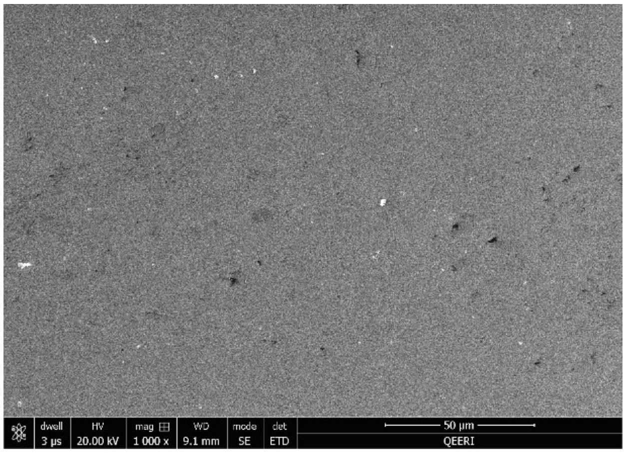

Figure 5. SEM of nickel oxide films at 10^{-4} Torr.

Scanning electron microscopy (SEM) analysis provides detailed insights into their surface morphology and microstructure. The SEM images typically reveal a uniform and dense morphology with well-defined grains, indicating successful deposition and a high-quality film. The grain size can vary based on deposition parameters, such as substrate temperature and oxygen flow rate, with optimized conditions leading to larger, more uniform grains that enhance the film's electrical and optical properties. The contact angle measurements of electron beam evaporated nickel oxide (NiO) thin films provide critical insights into their wettability and surface energy characteristics, which are essential for applications in optoelectronics and photovoltaics.

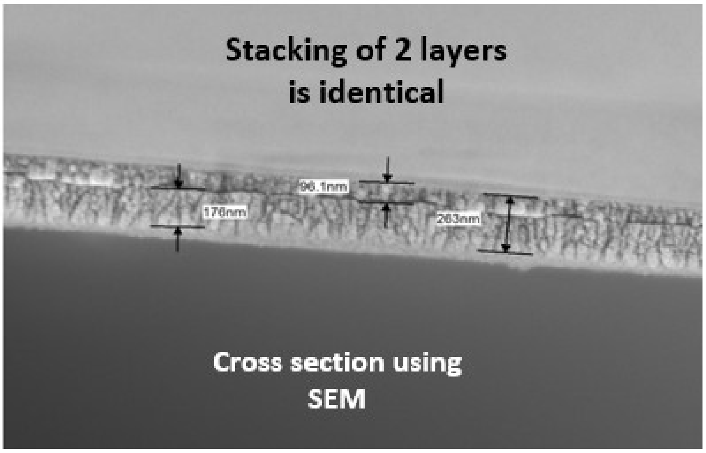

Figure 6. Cross section of nickel oxide films at 10^{-4} Torr.

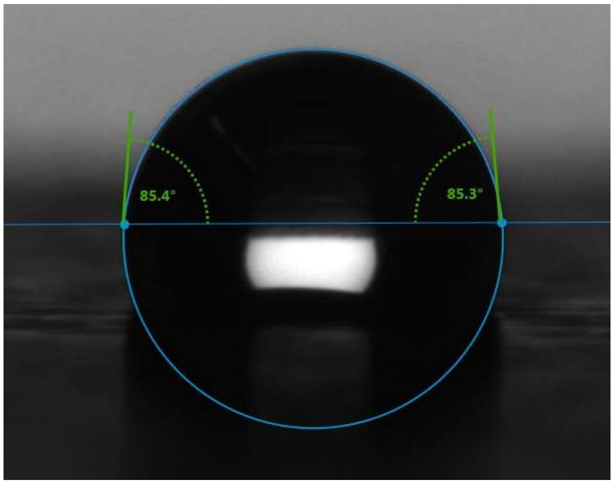

Figure 7. Contact angle measurement of nickel oxide films at 10^{-4} Torr.

The electrical properties of nickel oxide (NiO) thin films were evaluated using Hall effect measurements conducted at room temperature. The results indicated that the bulk carrier concentration of the films is approximately 10^{18} cm^{-3} for films deposited at 200 C. The highest mobility recorded was 12 cm^2/Vs for NiO films, while the lowest resistivity was also observed in films deposited at lower oxygen glow.

Table 2. This is a table. Tables should be placed in the main text near to the first time they are cited.

Deposition pressure (Torr)	Bulk Concentration [$\times 10^{18}$] ($/cm^3$)	Mobility [$\times 10^2$] (cm^2/Vs)	Resistivity [$\times 10^2$] (Ω-cm)	Hall Co-efficient [$\times 10^4$] (cm^3/c)
10^{-4}	2.6	6	3.4	0.5
10^{-5}	3.1	12	3.1	1.1

4. Discussion

The intensity and sharpness of these diffraction peaks can be influenced by the deposition parameters, such as substrate temperature, oxygen partial pressure, and post-deposition annealing conditions. A well-defined peak at (111) often indicates a preferential orientation, suggesting the formation of crystalline NiO. In some cases, broad or less intense peaks may imply the presence of nanocrystalline or amorphous phases, which can be further examined by adjusting the deposition

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settings. The XRD analysis not only confirms the phase composition of NiO but also provides information on grain size and crystalline quality, both of which are critical for optimizing the material's performance in optoelectronic and photovoltaic applications. This high transmittance is essential for efficient light harvesting when NiO is used as a hole transport layer in solar cells. The absorptance spectra, derived from the transmittance and reflectance data, highlights the film's ability to absorb light in the ultraviolet and near-infrared regions, which is influenced by the bandgap energy of NiO. The optical bandgap, estimated from Tauc plots, generally falls within the range of 3.5 to 4.0 eV, confirming the wide bandgap nature of NiO. These UV-Vis results indicate that NiO thin films exhibit desirable optical properties, such as high transparency and appropriate light absorption, which can be optimized for enhanced performance in energy conversion applications. Additionally, SEM results may show a smooth surface with minimal defects or voids, which is critical for applications in optoelectronics where surface quality directly influences device performance. The cross-sectional SEM images can further illustrate the film thickness and adherence to the substrate, confirming that the NiO layers are consistently deposited across the substrate surface. Overall, SEM characterization underscores the potential of e-beam evaporated NiO films for applications requiring high-quality thin films, such as hole transport layers in photovoltaic devices. ilms deposited at lower temperatures may experience tensile stress, resulting in smaller grain sizes and a more compact structure. In contrast, higher deposition temperatures can facilitate strain relaxation, altering the growth dynamics and impacting the final film thickness. The interaction between the sputtering target and the substrate is also temperature dependent. At elevated temperatures, increased energy can modify collision dynamics and influence deposition characteristics, ultimately affecting the overall thickness of the films. The rate at which atoms diffuse across the substrate surface is influenced by temperature; higher temperatures enable greater atomic mobility, which can enhance coalescence and lead to a more uniform film thickness. However, if temperatures are excessively high, there is a risk of re-evaporation or other material losses. Although the working pressure during sputtering remained constant in our experiments, it is important to note that variations in pressure can impact the mean free path of sputtered atoms and subsequently affect film growth characteristics. The thickness of sputtered tungsten (W) thin films varies with substrate temperature due to the complex interplay among atom mobility, film stress, surface diffusion, and sputtering dynamics. Each of these factors can either facilitate or hinder growth, resulting in the observed variations in thickness. The contact angles observed for these films typically indicate semi-hydrophilic properties, suggesting a moderate affinity for water. This semi-hydrophilic behavior is beneficial as it enhances the adhesion of subsequent layers, such as organic semiconductors or additional metal oxides, thereby improving the overall performance of devices. The measured contact angles can vary based on deposition parameters, including oxygen flow rate and substrate temperature, which influence the surface roughness and chemical composition of the films. A consistent trend observed is that films deposited with higher oxygen concentrations tend to exhibit lower contact angles, indicating improved wettability. These findings underscore the importance of optimizing the deposition conditions to tailor the surface properties of NiO films for enhanced functionality in various applications, including as hole transport layers in solar cells where effective charge carrier transport is crucial. Interestingly, as the deposition pressure decreases, the mobility tends to decrease. This decline in mobility is primarily attributed to the carrier-carrier scattering effect, which becomes more pronounced in films with higher bulk carrier concentrations, increasing the likelihood of collisions among carriers and consequently resulting in lower mobility. These findings highlight that variations in deposition temperatures significantly influence the electrical properties of the NiO films.

5. Conclusions

In this study, we employed a reactive electron beam evaporation technique to fabricate monolayers composed of metal oxide materials. Nickel oxide (NiO) layers exhibited impressive transmittance rates exceeding 80% in the visible spectrum. The findings indicate that the metal oxide layers produced through thermal e-beam evaporation can serve effectively as carrier transport materials, and being suitable for large-scale manufacturing. Extensive investigations have been carried out on the structural characteristics and the films demonstrated compactness throughout, as evidenced by scanning electron microscopy (SEM) images, and were devoid of pinholes, which is highly advantageous. The surface morphology of the films is significantly influenced by deposition pressure, with SEM images showing that a slower deposition rate results in a smoother surface. Optical measurements of samples produced under various conditions confirm the relationship between oxygen content in the films and their optical characteristics. Ongoing research is focused on precisely quantifying the impact of temperature variations and establishing correlations with diverse dust and weather conditions. Furthermore, future efforts should prioritize the design of devices that achieve optimal performance by carefully selecting hole transport materials (HTMs) with ideal optical properties while minimizing defects in both the bulk and interfaces. The optimized layers should be directly applied as carrier transport layers in large-scale solar cells.

Author Contributions: Mohammad Istiaque Hossain: Data curation, Formal analysis, Investigation, Methodology, Validation, Visualization, Roles/Writing - original draft.
Brahim Aissa: Supervision, Validation.

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Data Availability Statement: The data that support the findings of this study are available from the corresponding author, [MIH], upon reasonable request.

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

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