Tikrit Journal of Pure Science Vol. 31 (2) 2026

DOI:https://doi.org/10.25130/tjps.v31i2.1903

IRAQI Academic Scientific Journals

Tikrit Journal of Pure Science

ISSN: 1813–1662 (Print) --- E-ISSN: 2415–1726 (Online)

Journal Homepage: https://tjpsj.org/

Tikrit Journal of Pure Science

Synthesis of CdO and NiO-Based Sandwich Structures Using Pulsed Laser Deposition: Morphological and Optical Characteristics

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Received: 9 Feb. 2025 **Received in revised format:** 18 Apr. 2025 **Accepted:** 24 Apr. 2025

Final Proofreading: 13 Apr. 2026 **Available online:** 25 Apr. 2026

ABSTRACT

High-quality thin films of nickel oxide (NiO) and cadmium oxide (CdO) were deposited on porous silicon wafers and glass slides using pulsed laser deposition (PLD). Laser pulses at 1064 nm and 700 mJ were used at a low pressure of 10^{-3} Torr, followed by a 60 min annealing at 300 °C to stabilize the films. A porous silicon wafer was prepared using a (10 min) photochemical etching process, resulting in a surface with a favorable diffusion of the particle distribution. A scanning electron microscope (SEM) was used to analyze the distribution of CdO and NiO particles on the porous silicon surface at (200 pulses) and (300 pulses) for CdO and NiO, respectively. The results showed that increasing the pulse count improved the homogeneity and regularity of the particle distribution on the porous silicon surface, while simultaneously reducing the particle size. The etching process facilitated surface dispersion and the formation of a homogeneous nanolayer on the porous silicon films, as evidenced by the photoluminescence (PL) spectra of CdO and NiO. UV-Vis analysis revealed that CdO and NiO deposited on glass slides exhibit strong absorption at short wavelengths, with film thickness amplifying this absorption.

Keywords: CdO, laser pulses, NiO, PLD, thin film thickness.

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تحضير تراكيب ساندويتش قائمة على أوكسيد الكادميوم وأوكسيد النيكل باستخدام الترسيب بالليزر النبضي: الخصائص المورفولوجية والبصرية

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قسم الفيزياء، كلية العلوم، جامعة تكريت، تكريت، العراق

الملخص

تم تحضير أغشية رقيقة عالية الجودة من أكسيد النيكل وأكسيد الكادميوم على شرائح السيليكون المسامي وشرائح الزجاج باستخدام تقنية الترسيب بالليزر النبضي (PLD). إذ استُخدمت نبضات ليزر بطول موجي (1064 nm) وطاقة نبضة تبلغ (700 mJ) تحت ضغط منخفض مقداره (10^{-3} Torr)، تلتها عملية تلدين لمدة (60 min) عند (300 °C) لتثبيت الغشاء. تم تحضير رقاقة سيليكون مسامية باستخدام عملية نقش ضوئي كيميائي استغرقت (10 min)، مما أدى إلى تكوين سطح يوفر انتشاراً ملائماً لتوزيع الجسيمات. وقد استخدم المجهر الإلكتروني الماسح (SEM) لتحليل توزيع جسيمات CdO وNiO على سطح السيليكون المسامي عند (200 pulses) و(300 pulses) لـ CdO وNiO على التوالي، حيث أظهرت النتائج أن زيادة عدد النبضات أدى إلى تحسين تجانس وانتظام توزيع الجسيمات على سطح السيليكون المسامي، مع تقليل حجم الجسيمات في نفس الوقت. ساعدت عملية الحفر على تشتت السطح وتطور طبقة نانوية متجانسة على الأغشية السيليكونية المسامية، كما هو موضح بواسطة طيف PL لـ CdO وNiO. كشف تحليل الأشعة فوق البنفسجية–المرئية أن CdO وNiO المترسبة على شرائح الزجاج يظهران امتصاصًا قويًا عند الأطوال الموجية القصيرة، حيث يؤدي سُمك الغشاء دورًا في تضخيم هذا الامتصاص.

INTRODUCTION

Depending on how the solid target interacts with the laser beam, PLD technology has been successfully used on numerous materials. Compared with more conventional physical vapor deposition techniques, this method offers several clear benefits, especially when using pulsed lasers. This method, which is performed in a gas-free or vacuum-suspended environment, produces thin films that, for most chemicals, are quite similar to the substance of interest (1-3). PLD makes an "ablation shaft" of particles that land on the base by excising a target material in the chamber with a powerful laser beam; this technique has been used for 25 years and is now considered the best for growing thin films (4, 5). A film is a thin, two-dimensional substance grown on a substrate by successively condensing the iconic, molecular, and atomic forms of matter; this method is commonly used for film fabrication. Films with a thickness of less than a few microns are common. Actually, there's another kind of film called a thick film. It's a two-dimensional material created by either melting a three-dimensional substance or by building up large grains, aggregations, or collections of iconic molecular or atomic species. Thick films are used to make hard coatings, decorative components, and electronic devices(6). An n-type semiconductor material with a straight wideband gap (2.2–2.7 eV) is CdO. Among their numerous uses, CdO materials are most often found in solar cell production due to their unique optical characteristics, such as a high transmittance in the visible range and relatively high electrical conductivity (7-9). It should also be noted that CdO has a very high optical transmission of about 90% in the visible part of the solar spectrum.

Additionally, the electrical conductivity of CdO thin films is quite impressive. Consequently, it has great potential for a range of applications, including gas sensors, organic light-emitting diodes, and flat-panel displays (10-12).

A metal-deficient p-type semiconductor with a band gap of 3.6 eV, NiO is renowned for its ferromagnetic properties (13). Films made of NiO are very versatile because they withstand chemicals well. One application for them is as catalysts(14-16). This study examined the impact of pulsed laser pulse count on the surface morphology and optical characteristics of thin films of CdO and NiO, and the effect of photoelectrochemical etching and thermal annealing. Additionally, the effects of film thickness on the absorbance and transmittance spectra were examined.

EXPERIMENTAL WORK

A ball mill to grind 99.9% pure CdO and NiO powders from Areej Al-Furat Company was used. These powders were then compressed into (2 g) tablets with a (10 mm) diameter and a (4 mm) thickness in a stainless-steel mold using a hydraulic press set at (5 tons) for (4 min). Using PLD in a vacuum chamber at 10^{-3} Torr, pre-cleaned glass substrates were coated with thin layers of CdO and NiO. With a 2.5 cm gap between the chip and the target and a 30 cm gap between the laser and the target, the laser beam was focused to a specific point on the target's surface at 45 °. A pulsed Nd: YAG laser was used for the deposition process; this laser has a fundamental wavelength of 1064 nm, a pulse duration of 10 ns, and varying pulse counts (200 and 300 pulses) of constant energy (700 mJ). For 60 min, the material was heated to 300 °C in an annealing oven. PL spectroscopy and SEM were used to ascertain the thin films' morphological characteristics. For this experiment, cross-sectional scanning electron microscopy was used to determine the film thickness. NiO thin films had a thickness of 430 nm, and CdO thin films of 840 nm. A porous silicon (P-Si) structure was prepared by electrochemical etching of a P-type silicon wafer (111) orientation and (0.01 Ω.cm) resistivity, in a Teflon cell, using a two-electrode configuration with a platinum counter electrode and a silicon wafer anode. The etching process took 10 min at 10 mA. Dilute hydrofluoric (HF) acid (1:10) with a diode laser source was used as the illumination source. The process involves annealing at 300 °C for 60 minutes and using variable pulse numbers (300 pulses) for CdO and 200 pulses for NiO.

RESULTS AND DISCUSSION

Morphological Properties

In Figures (1) and (2), the SEM analysis of the prepared thin films was conducted to identify the surface morphology and nanoparticle size. Figure 1 shows that the granules and particles formed on the surface of porous silicon doped with CdO have a size range of 92.32 to 31.10 nm. The particles appear to be clumped, with both larger and smaller particles present. This suggests that the particle distribution on the surface is nonuniform, with some areas having a high particle density and others a lower one. As a result, the porous surface has an uneven layer of CdO deposited by a pulsed laser. Figure 2 shows that NiO nanoparticles have a nearly spherical shape and a size range of 43.7 to 27.15 nm. This indicates that the deposited material consists of minuscule, tightly compacted granules. The deposition exhibited greater density and uniformity alongside an increase in pulse count corresponding to a drop in pulse count. The annealing process was essential for improving the crystallization and so augmenting the material's chemical stability. This result indicates that increasing the laser pulse count from 200 to 300 enhanced the homogeneity and distribution of the NiO nanoparticles, while decreasing their size relative to that of CdO nanoparticles.

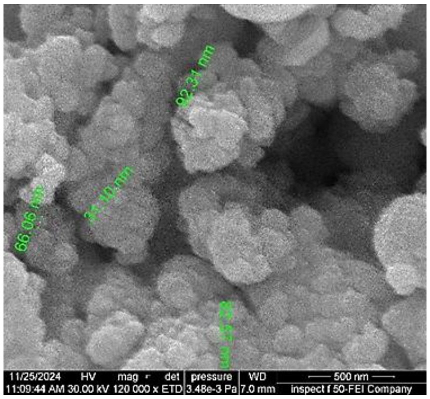

Fig. 1: 3D image for the surface of CdO nanofilms analyzed by SEM test at a time of engraving (10 minutes) and 200 pulses.

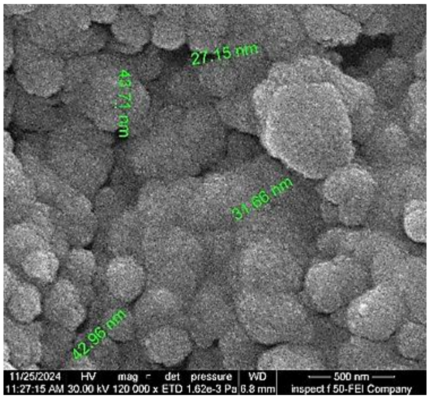

Fig. 2: 3D image for the surface of NiO nanofilms analyzed by the SEM test at the time of engraving (10 minutes and 300 pulses).

The larger and less regular particles of CdO may stem from a difference in crystal growth rate or material properties during evaporation and re-deposition. Additionally, NiO possesses good electrochemical properties, making this structure suitable for gas sensors and solar cells (17), particularly due to its high conductivity and stability under varying environmental conditions. Figure 3 shows a scanning electron micrograph (SEM, cross-sectional) indicating that the film has a thickness of 398.1-446.3 nm. NiO was efficiently and uniformly deposited onto glass slides, as evidenced by a regular surface structure and good particle dispersion. Since the PLD method helped regulate the deposition rate, the uniform surface structure indicates a crystallization process. The nanofilm enhances optical efficiency, and its thickness is within the appropriate range for use in optical and electronic devices, including photodetectors and solar cells. Thermal annealing decreased distortions caused by deposition and increased surface uniformity. In light of the foregoing, we infer that the film's unique structural characteristics result from the NiO material's efficient interaction with the glass substrate. Because of these qualities, the material can be used in industrial applications that require materials with excellent optical and mechanical properties, such as the production of high-performance optical devices and durable coatings for various surfaces.

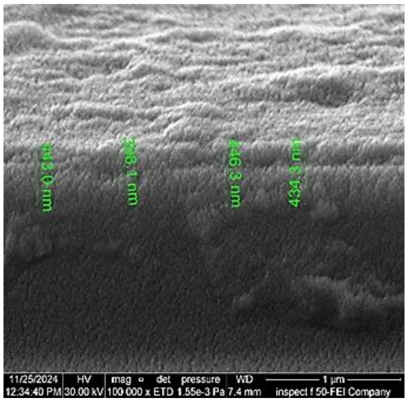

Fig. 3: SEM micrographs (cross-sectional and top-surface morphology) obtained for NiO thin films on the glass.

Optical Properties

The UV-Vis spectrophotometer was used to obtain optical spectra, including absorbance and transmittance. PLD of CdO onto a glass slide is shown in Figure 4 as an optical absorbance spectrum. The material's absorbance is highest in the visible spectrum (200–800 nm) and gradually decreases as one moves farther into the infrared spectrum, as shown in the absorbance curve plotted against wavelength. Applications in photovoltaic devices, such as solar cells and photodetectors, benefit from the material's strong absorption in this range, demonstrating its efficient absorption of visible light. The film's thickness affected the absorbance. Light was absorbed more rapidly in thicker films. Thermal annealing strengthened the material's crystalline structure and reduced internal flaws, thereby stabilizing its absorbance in the visible range. Applications that solely require optical stability in the visible range will consider this material suitable, as its gradual decrease in absorbance at longer wavelengths indicates its ability to reduce the absorption of infrared radiation. These findings demonstrated that CdO is a technologically advanced material with unique optical characteristics, making it ideal for use in photoelectric and optical devices.

The optical absorbance spectra of NiO are displayed in Figure 5. The high absorption at short wavelengths (300–400 nm) in the spectrum results from photons with energies consistent with nickel oxide's energy gap, which reveals electronic transitions between the valence and conduction bands. The absorbance drops off when the wavelength rises above 400 nm because the photons lose some of their energy and can no longer excite electrons. At shorter wavelengths (430 nm), increasing the film thickness increased absorbance; however, as wavelengths increased, absorbance decreased because photons interacted with the material less effectively. By increasing the crystal structure and reducing flaws in the film, the absorbance properties were improved, and losses from non-radiative processes were reduced through thermal annealing. The spectra show that NiO is a semiconductor with a broad bandgap, which makes it useful for optical and photovoltaic applications such as solar cells, light sensors, and light-absorbing coatings. NiO is used in many technological applications due to its strong visible-spectrum absorption, which gives it these properties.

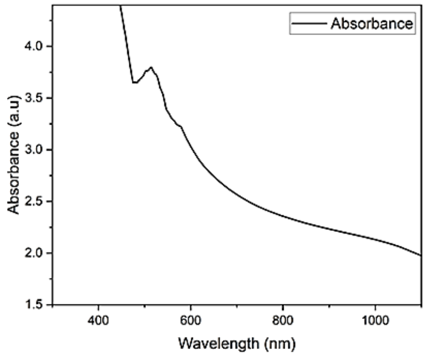

Fig. 4: UV-visible absorption spectrum of CdO thin films.

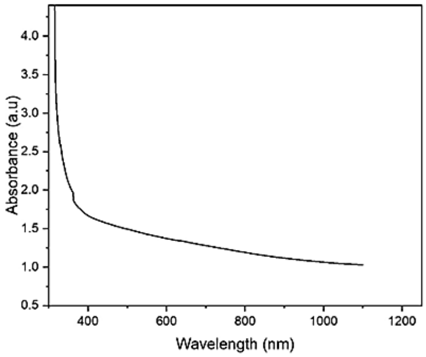

Fig. 5: UV-visible absorption spectrum of NiO thin films.

CdO thin film optical transmittance spectra shown in Figure 6 were acquired. Low transmittance values are seen at short wavelengths (600 nm) in the spectrum, which rise steadily to a peak at 1200 nm as the wavelength increases. CdO has a small energy gap, so its transmittance gradually increases. This is because the material absorbs high-energy photons (short wavelengths) and transmits low-energy photons (long wavelengths). The low pressure used during deposition reduced flaws and impurities, enhancing the film's quality. Optical transmittance was enhanced by its role in improving crystal structure and reducing imperfections. The transmittance was reduced at short wavelengths due to absorption and scattering, as the film thickness was quite considerable at 840 nm, although this effect became less apparent at longer wavelengths. Based on the information provided, it can be inferred that CdO thin films possess favorable optical properties. These properties render them well-suited for a range of applications, including solar cells, optical sensors, and anti-reflection coatings. Depending on the specific requirements of each application, these films can achieve high levels of light absorption or transmission efficiency.

The optical transmittance spectrum of the NiO thin film is studied in Figure 7. The high absorption at short wavelengths (300–400 nm) in the spectrum of the prepared films exhibits an optical transmission of 9%. This is due to photons with energies consistent with the NiO band gap. The absorption decreases as the wavelength rises above 400 nm because the photons lose some of their energy and are no longer able to excite electrons. At shorter wavelengths, as wavelengths increase, the transmittance increases because the photons interact less effectively with the material.

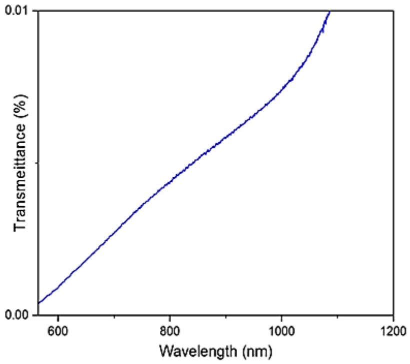

Fig. 6: UV-visible transmittance spectrum of cadmium oxide (CdO).

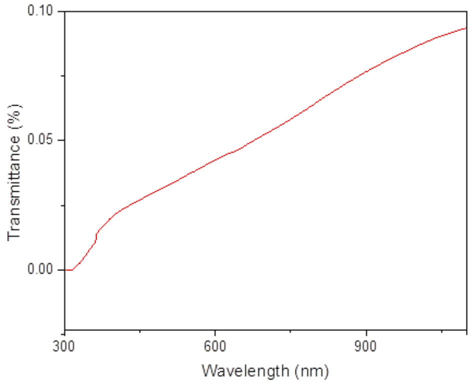

Fig. 7: UV-visible transmittance spectrum of nickel oxide (NiO).

PL spectra

Figure 8 displays the results of PL of CdO placed on a porous silicon wafer using a pulsed laser approach. The emission spectra depicted show a primary peak at 438.8 nm, which falls within the blue spectral region. Since the formed material displays an optical energy gap (Eg) of 2.82 eV, calculated using equation (1), this peak suggests high-energy electronic transitions [(18)].

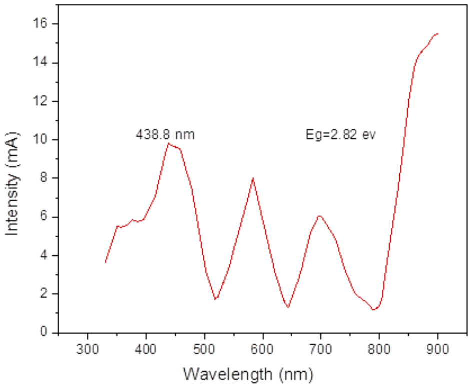

Fig. 8: PL spectra of CdO/PSi nanostructures prepared with a 10-minute etching time and 200 pulses.

By creating deep pores through etching, the porous structure had a greater impact on emission properties, and the regularity of the deposited surface was enhanced due to improved interaction between the deposited layer and the porous surface. The thermal annealing procedure greatly enhanced both surface uniformity and interfacial defect reduction. For high-power applications such as light-emitting diodes (LEDs), blue emission is ideal because it exhibits high-energy characteristics.

PL data shown in Figure 9 indicate that the deposited NiO exhibits unique optical properties, with a primary emission peak at 433.6 nm. The calculated Eg of 2.86 eV indicates that the deposited material exhibits good semiconductor properties and is suitable for optical and electrical device applications. Surface regularity and interfacial defect reduction were both significantly enhanced by thermal annealing. The electrochemical etching process, which formed deeper pores, enhanced the emission properties of the porous silicon laminates. The sedimentation layer was made more homogeneous and of higher quality thanks to the low pressure used during the process. This variation in the energy band gaps of NiO and CdO is caused by the interplay between quantum confinement and enhanced surface states, which alters the materials' electronic structures and widens the energy gaps (19, 20).

$$Eg = \frac{hc}{\lambda} = \frac{1240}{\lambda(nm)} \qquad \dots (1)$$

The values of Planck's constant (h) and velocity of light in a vacuum (c) are (6.62×10^{-34} J/s) and (3×10^{8} m/s), respectively.

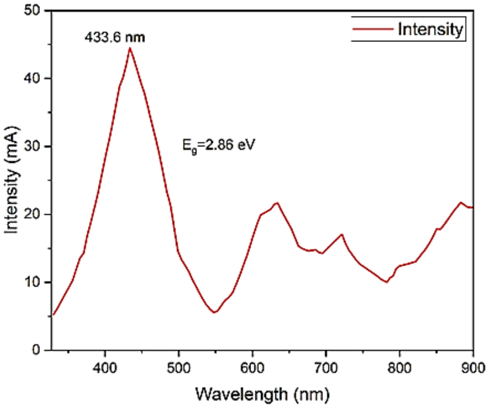

Fig. 9: PL spectra of NiO/PSi nanostructures prepared (10 minutes) etching time and (300 pulses).

CONCLUSION

This study demonstrated that PLD is an effective method for depositing thin films of CdO and NiO on porous silicon substrates. Spectroscopic analyses revealed optical band gaps of (2.86 eV) for NiO and (2.82 eV) for CdO, indicating that these materials possess suitable optical properties for optical and electronic applications. SEM results also demonstrated uniform deposition of the layers with heterogeneous particle distributions, with NiO having nearly spherical particles and CdO having agglomerated particles. These results confirm the potential of the prepared materials in advanced applications such as solar cells and sensors.

Conflict of interest: The authors declared no conflicts of interest.

Sources of funding: This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Author contributions: The authors contributed equally to the study.

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