Growth and Characterization of Multilayer NiO/Ag and NiO/Al Structures for Energy Saving Heat Mirrors

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Optically Transparent multilayer NiO/Ag and NiO/Al heat mirrors were prepared, which allow the visible radiation to pass through them and reflect the Infrared radiation. NiO thin films were deposited by the sol-gel spin coating technique, and Ag and Al thin films were prepared by thermal evaporation. XRD analysis showed the formation of a cubic structure of NiO. The chemical analysis reveals that the Ni_2O_3 phase is also present along with the NiO phase. The transmittance spectra of NiO/Ag and NiO/Al coatings showed good transmittance in the visible region, while being highly reflective in the infrared region. XRD analysis of the film showed the formation of a cubic structure of NiO. The chemical analysis showed that some peaks belong to another phase of the NiO. The transmittance spectra of multilayers of NiO/Ag and NiO/Al films are transparent in the visible region and reflect well in the infrared region.

Keywords: Optically transparent coatings, NiO/Ag multilayer films, NiO/Al multilayer films, Sol-gel spin coating, Chemical composition analysis

Introduction:

Global energy consumption is rising steadily, leading to an increased demand for energy resources. Buildings account for approximately 40% of total energy use, primarily due to lighting, heating, and cooling needs [1]. The extensive use of cooling and heating systems in both hot and cold regions has resulted in a significant increase in electricity consumption to regulate indoor temperatures. According to the World Energy Council, 62 % of energy is generated from oil and coal [2]. The energy production from fossil fuels generates carbon dioxide and other pollutant gases that lead to an increase in the global warming issue. To minimize the usage of energy to decrease the production of polluted gases, which are harmful to both our environment and health [3]. Heat mirror insulated transparent windows allow the transmission of 98% of the visible light and reflect 90% of the infrared radiation [4]. The use of heat mirror technology is an accepted means of energy conservation. Heat mirrors are thin coatings that allow visible light to pass through them and block all other radiation. It is very important to use a heat mirror for energy-saving windows. Nowadays, plain glass slides are commonly used as windows in buildings, and these windows are transparent to both visible and infrared radiation. Infrared radiations are also known as heat radiations that actually enter in room in summer from outside and affect the temperature of the room [5]. To maintain the desired temperature of the room, a window is required that allows visible light to pass through it and blocks other radiation. For optimal performance, windows must be transparent to the solar spectrum and simultaneously minimize all forms of thermal loss. One way to overcome thermal losses while maintaining high solar transmission is the use of heat mirrors [6]. Nickel oxide (NiO) is a transition metal oxide with a cubic lattice structure. It is a highly important material due to its wide range of applications, including electrochromic films, gas sensors, catalysis, dye-sensitized photocathodes, and magnetic materials [7]. It was also reported in literature that nano-sized NiO particles possess much improved properties compared to micrometer-sized NiO particles. Hollow Nano sphere of NiO nanospheres are used as a glucose sensor. The solar radiation spectrum range is from 380 nm to 3000nm [8]. The radiation below 400 nm is UV radiation, from 400 nm to 700 nm range radiations are visible radiations, and from above 700 nm, the radiations are known as infrared radiation [9]. Energy-efficient windows are designed in such a way that pass only visible light to the interior and reflect the infrared radiation to the exterior. For such a type of window, we need a transparent coating that allows visible light to pass through it and reflects the infrared light into the exterior. Multilayer structures based on metal and dielectric materials that allow visible light to pass through them and block heat radiation (infrared radiation) [10]. These structures are commonly referred to as Transparent Heat Mirrors (THMs).

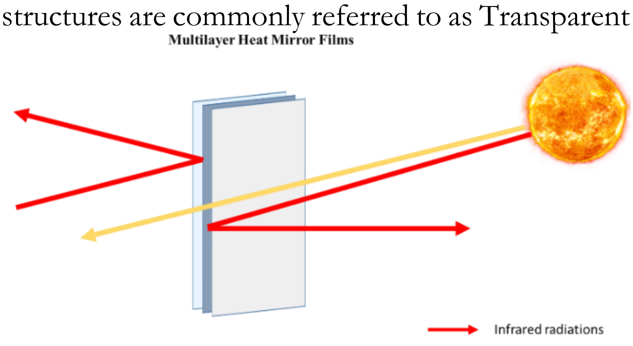

Figure 1. Schematic diagram of HEAT Mirror

Objectives:

1. To synthesize and fabricate multilayer NiO/Ag and NiO/Al thin films using sol-gel spin coating and thermal evaporation techniques for potential use in transparent heat mirror applications.
2. To characterize the structural, chemical, and optical properties of the prepared NiO/Ag and NiO/Al multilayers using XRD, XPS, and UV-Visible/NIR spectrophotometry.

3. To evaluate and compare the transmittance in the visible spectrum and reflectance in the infrared region of NiO/Ag and NiO/Al multilayer structures to assess their efficiency in minimizing thermal losses through window glass.

Literature Review:

The energy crisis is becoming a serious problem, leading researchers to develop some new techniques and products that consume minimal energy to overcome the energy crisis. One of the lamp manufacturers starts working with researchers at the Massachusetts Institute of Technology (MIT) to develop new and energy-efficient incandescent lamps [11]. In 1970, the energy crisis became a serious problem, leading researchers to develop some new techniques and products that consume minimal energy to overcome the energy crisis [12]. According to the literature, an increase in filament temperature leads to an improvement in its efficiency. The filament emits energy in the form of infrared rays that are invisible but we feel as heat. If the emitted heat comes back towards the filament and increases the temperature of the filament, the bulb gives more light with no need for excess power. Researchers named this concept "heat-mirror". They introduce a transparent thin film coating, which allows visible light to pass through it and reflect infrared back into the filament, and insert it inside the glass bulb [13].

Author defined a heat mirror that it is a coating that can selectively transmit or reflect a portion of light, which shows suitable reflection or transmissions of the electromagnetic spectrum in three ranges of radiant energy. The three ranges included the visible 300nm to 700nm, near-infrared (NIR), 700-2000 nm, and infrared (IR), 2.0 nm, and above. Heat mirrors show high transmittance in the visible and low emittance throughout the infrared range [13][14].

Authors prepared a multi-layer transparent film that consists of $Ti0_2$/ Ag/$Ti0_2$ on a glass substrate of CG7059 for solar energy applications by the RF sputtering technique. They mentioned in their results that this type of structure exhibits excellent transmission in the visible region and has good reflectivity in the infrared region. This type of film exhibits superior thermal and chemical stability, making it highly suitable for use in thermal insulating coatings for both residential and commercial buildings. However, the main challenges related to these films are stability and durability [15].

Methodology:

To prepare Bi-layer NiO/Ag and NiO/Al heat mirrors, NiO films were deposited by the Sol-Gel spin coating Process, and Ag and Al films were deposited by thermal evaporation. For the preparation of NiO film, dissolve Nickel nitrate hexahydrate [$Ni (NO_3)_2.6H_2O$, Sigma 99.9%] into 20 ml of an equal amount of isopropanol alcohol (Sigma 99.7 %) and polyethylene glycol 200 [$H (OCH_2CH_2)nOH$ (sigma)] to make a 0.1 M solution. Then the solution was stirred using a magnetic stirrer at 50° C for 3 hours to get a transparent solution. Dilute ammonium hydroxide, [NH_4OH], is added dropwise to the solution under magnetic stirring nickel hydroxide colloid is produced. To prevent the agglomeration of particles, Triton X-100 was added dropwise to the solution, effectively lowering the surface tension of the NiO particles. After the above process, the solution was stirred for one hour before coating. The glass substrate was used to deposit the NiO film. Before film deposition, the glass substrate was thoroughly cleaned using acetone, followed by distilled water to eliminate any dust particles and organic contaminants. After that films were annealed at 400 °C for 3 h in a muffle furnace. Silver and Aluminum thin film was deposited through the thermal evaporation technique in a vacuum environment.

A small piece of pure silver metal was placed on the molybdenum board, and the glass substrate was secured onto the substrate holder. The distance between the silver source and the substrate was maintained at 8 cm. A Vacuum environment was set at 3.5×10^{-5} mbar to obtain a fine film. Start to increase the current, and at 78 to 80 amps, silver metal starts to evaporate. To obtain a transparent film, allow the evaporation for a time duration of 40,45,50,55 sec for different films of thickness. The same procedure was followed for the deposition of the aluminum thin film. After the deposition of the metal film, NiO film was coated using a spin coating technique at a rotational speed of 3000 rpm for 10 seconds. The prepared films were preheated at 120°C for 1 hour to remove solvents after coating each layer. The spin coating process was repeated multiple times to achieve the desired thickness of the NiO film. At the end multilayer transparent NiO/Ag film and a NiO/Al film were prepared.

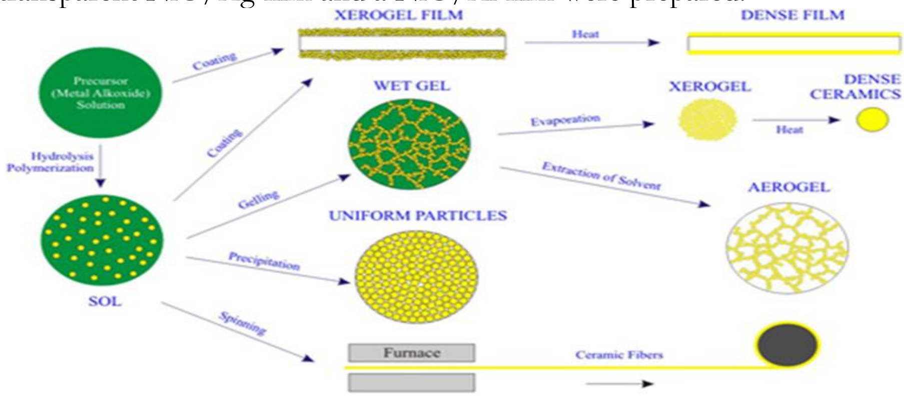

Figure 2. Flow Diagram of Methodology

XRD was used to determine the structural characteristics of the materials. The crystallite size was calculated by Scherer's formula [16],

$$D = \frac{k\lambda}{\beta Cos\theta} \qquad (1)$$

In equation (1), D represents particle size, λ is the wavelength, θ is the diffraction angle, k is a constant, and β= FWHM (Full width half maximum). The average crystallite size is 22 nm. The chemical analysis was studied using XPS. This approach detects core-level variations and offers information about the metal's surroundings and oxidation state. The optical transmittance was utilized to determine the band gap of a thin film (Eg). In the optical absorption region, the transmittance of the film is expressed as $T = e^{-\alpha d}$. Therefore, the absorption coefficient can be written as [17]:

$$\alpha = \frac{1}{d}\ln(\frac{1}{T}) \qquad (2)$$

Here, α is the absorption coefficient, T is the transmittance, and d is the film thickness. The thickness of the film was measured with a surface profilometer. The band gap of the NiO film was determined using the following equation.

$$(E\alpha)^{1/Z} = \beta\,(E - E_g) \qquad (3)$$

Where β is a constant, E is the photon energy, and z is the power factor of a transition mode, and thus z = 2, which depends on the nature of the material.

Results and Discussion:

Structural analysis:

Figure 3 represents the XRD pattern of the NiO film. Distinct diffraction peaks corresponding to the (111), (200), (220), (311), and (222) crystal planes indicated by (*) appeared at 2θ angles of 37.41°, 43.45°, 62.98°, 75.50°, and 79.40°, respectively. XRD result revealed that the NiO film is polycrystalline and the preferred orientation is along the 200-crystal plane. These diffraction peaks were perfectly matched with the JCPDS file no 04-0835 [18]. It was observed that an impurity peak marked with (square box) was found at 44.66, which belongs to the second phase of Ni_2O_3.

Chemical Analysis:

Chemical analysis was carried out using X-ray Photoelectron Spectroscopy (XPS), a technique highly sensitive to core-level shifts. It provides valuable insights into the chemical environment of the metal and its oxidation state. Figure 4 represents the Ni 2p core level spectrum of the annealed NiO film. It was observed that the Ni 2p core level spectrum consists of two sublevels (2p1/2 and 2p2/3) because of spin-orbit coupling. All the recorded spectra were analyzed using Avantage Software. A standard carbon peak, C 1s, was observed at 284.80 eV in the spectrum and was used as the reference peak for calibrating all other spectra. To get the complete information from the XPS spectra, a standard Shirley-type background correction was carried out. The spectra were deconvoluted and fitted using a Lorentzian-Gaussian line shape to determine the chemical composition of the NiO film. Each 2p $_{3/2}$ and 2p $_{1/2}$ was resolved into two peaks that represent the two oxidation states of Ni, as shown in the figure 4. Similarly, the Oxygen peak was also deconvoluted into two peaks. A peak at 530 eV represents the NiO phase, while a peak at 532.00 eV represents the Ni_2O_3 phase. The chemical analysis results were consistent with previous XRD patterns, confirming the presence of both phases within the film.

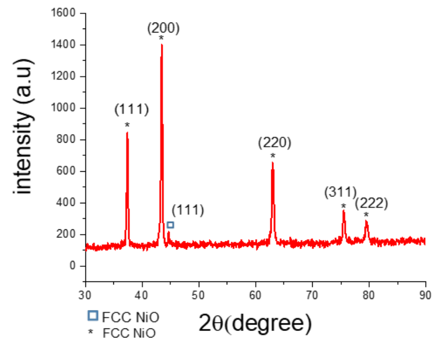

Figure 3. XRD pattern of annealed NiO film

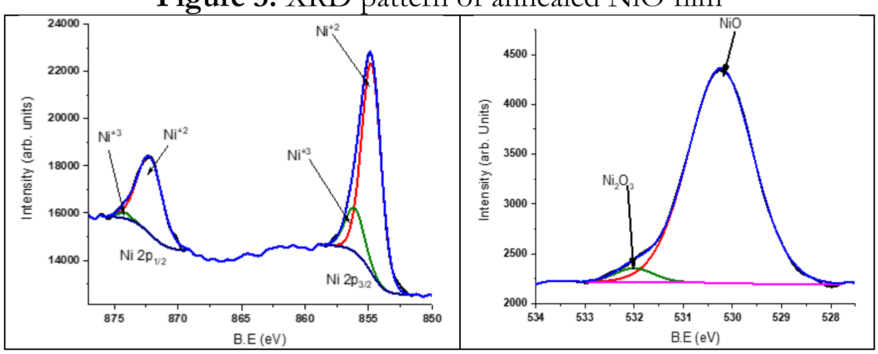

Figure 4. XPS Ni 2p core level spectra of annealed NiO film

Optical Properties:

Transmittance of NiO film:

Figure 5 shows the transmittance of the NiO film. It was observed that the NiO film is highly transparent in the visible and infrared regions. With the help of a spectrophotometer model (Jasco V-770 UV-Visible/NIR spectrophotometer) individually measured the optical properties of NiO, Ag, and aluminum thin films. The wavelength of the spectrophotometer was adjusted from 1- 2000 nm to measure the optical properties of the films.

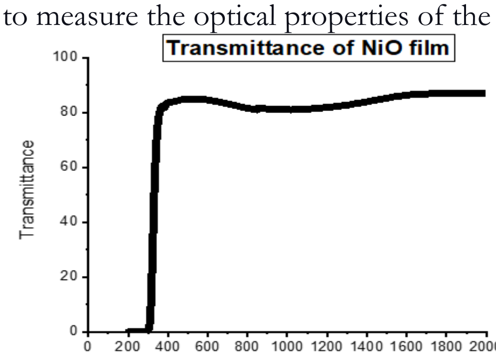

Figure 5. Transmittance of annealed NiO film

Transmittance of Ag film:

As shown in Figure 6, the silver film exhibits high reflectance in the infrared region, exceeding 70%, while maintaining a visible light transmittance of up to 65%. At wavelengths below 300 nm, the film exhibits high absorption, effectively blocking any light from passing through.

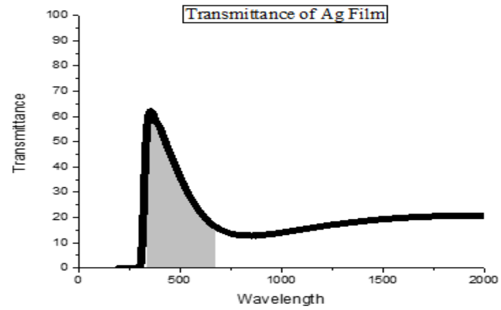

Figure 6. Transmittance of Ag Film

Transparent Heat Mirror:

Figures 7 (a) and 7 (b) represent the transmittance spectra of NiO/Ag and NiO/Al thin films, respectively. It was observed that NiO/Al film showed maximum transmittance 68 % at 465 nm and NiO/Ag film showed maximum transmittance 71% at 632 nm. Overall, both films showed transmittance greater than 55 % in the complete visible region and were highly reflective in the infrared region. These coatings are expected to be used as energy-saving window materials in the future, due to their high transmittance in the visible region and strong reflectance in the infrared region, which helps minimize energy losses.

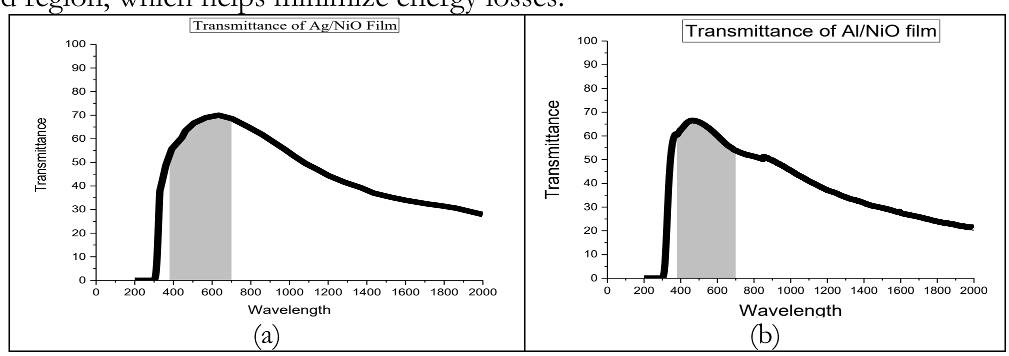

Figure 7. (a) Transmittance of NiO/Ag and (b) Transmittance of NiO/Al

Determination of the band gap of NiO film:

The optical transmittance was used to calculate the band gap of the thin film (E_g). Fig. 8 represents plots of (αE)1/2 as a function of photon energy (E). The extrapolation of the linear portion provided the band gap value of the film.

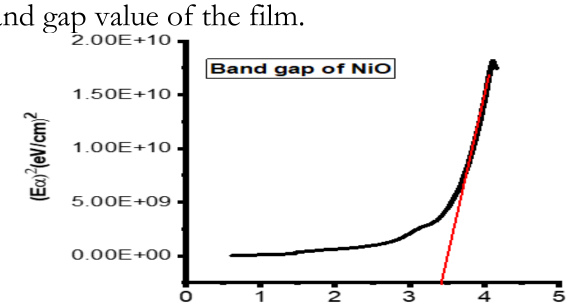

Figure 8. Band Gap of NiO by Tauc Plot

Conclusion:

NiO/Ag and NiO/Al coatings were fabricated successfully by sol sol-gel spin coating technique and thermal evaporation. NiO film was deposited by sol-gel spin coating, and Ag and Al films were prepared by thermal evaporation. The XRD result showed the formation of a cubic structure of NiO. It was also noticed that a peak observed at 44.66 belongs to the second phase of Nickel oxide, Ni_2O_3. The crystallite size was estimated by the Scherrer formula, and the estimated value of the crystallite size is 22 nm. The chemical analysis revealed that two oxidation states, Ni^{+3} and Ni^{+2}, of Ni exist in the NiO film, which indicates the presence of two phases of Nickel oxide. This result is consistent with the XRD result. The transmittance of NiO films showed that it is highly transparent in the visible and infrared regions. The band gap of the NiO film was measured by the Tauc plot, and the estimated value of the band gap is 3.4 eV. The transmittance spectrum of NiO/Ag film showed that it showed good transmittance in the visible region and had very little transmittance in the infrared region. NiO/Ag film showed maximum transmittance 71% at 632 nm. Similarly, NiO/Al film also showed good transmittance in the visible region and has a maximum transmittance 68 % at 465 nm. Therefore, these coatings will be used in the future as windows to save energy consumption.

References:

[1] X. Cao, X. Dai, and J. Liu, "Building energy-consumption status worldwide and the state-of-the-art technologies for zero-energy buildings during the past decade," *Energy Build.*, vol. 128, pp. 198–213, 2016, doi: https://doi.org/10.1016/j.enbuild.2016.06.089.

[2] W. H. Haider, "Estimates of Total Oil & Gas Reserves in The World, Future of Oil and Gas Companies and SMART Investments by E & P Companies in Renewable Energy Sources for Future Energy Needs," *Int. Pet. Technol. Conf. 2020, IPTC 2020*, Jan. 2020, doi: 10.2523/IPTC-19729-MS.

[3] I. Manisalidis, E. Stavropoulou, A. Stavropoulos, and E. Bezirtzoglou, "Environmental and Health Impacts of Air Pollution: A Review," *Front. Public Heal.*, vol. 8, p. 505570, Feb. 2020, doi: 10.3389/FPUBH.2020.00014/BIBTEX.

[4] C. M. Chen, S. Y. Chen, W. C. Chuang, and J. Y. Shieh, "Transparent Glass Window with Energy-Saving and Heat Insulation Capabilities," *Adv. Mater. Res.*, vol. 314–316, pp. 10–16, 2011, doi: 10.4028/WWW.SCIENTIFIC.NET/AMR.314-316.10.

[5] Marco Casini, "Active dynamic windows for buildings: A review," *Renew. Energy*, vol. 119, pp. 923–934, 2018, doi: https://doi.org/10.1016/j.renene.2017.12.049.

[6] B. L. Yu Qiu, Yucong Xu, Qing Li, Jikang Wang, Qiliang Wang, "Efficiency enhancement of a solar trough collector by combining solar and hot mirrors," *Appl. Energy*, vol. 299, p. 117290, 2021, doi: https://doi.org/10.1016/j.apenergy.2021.117290.

[7] S. H. M. Suhas H. Sutar, Bapuso M. Babar, Komal B. Pisal, Akbar I. Inamdar, "Feasibility of nickel oxide as a smart electrochromic supercapacitor device: A review," *J. Energy Storage*, vol. 73, p. 109035, 2023, doi: https://doi.org/10.1016/j.est.2023.109035.

[8] X. Huang *et al.*, "Graphene-Based Materials: Synthesis, Characterization, Properties, and Applications," *Small*, vol. 7, no. 14, pp. 1876–1902, Jul. 2011, doi: 10.1002/SMLL.201002009.

[9] D. H. Sliney, M. Bitran, and W. Murray, "Infrared, Visible, and Ultraviolet Radiation," *Patty's Toxicol.*, pp. 169–208, Aug. 2012, doi: 10.1002/0471435139.TOX102.PUB2.

[10] H. S. Charu Dwivedi, Priyanka Bamola, Bharti Singh, "Chapter 2 - Infrared radiation and materials interaction: Active, passive, transparent, and opaque coatings," *Energy Sav. Coat. Mater.*, pp. 33–56, 2020, doi: https://doi.org/10.1016/B978-0-12-822103-7.00002-9.

[11] M. Kavehrad, "Sustainable energy-efficient wireless applications using light," *IEEE Commun. Mag.*, vol. 48, no. 12, pp. 66–73, Dec. 2010, doi: 10.1109/MCOM.2010.5673074.

[12] N. Armaroli and V. Balzani, "The Future of Energy Supply: Challenges and Opportunities," *Angew. Chemie Int. Ed.*, vol. 46, no. 1–2, pp. 52–66, Jan. 2007, doi: 10.1002/ANIE.200602373.

[13] Carl M. Lampert, "Heat mirror coatings for energy conserving windows," *Sol. Energy Mater. Sol. Cells*, vol. 6, no. 1, pp. 1–41, 1981, doi: https://doi.org/10.1016/0165-1633(81)90047-2.

[14] Carl M. Lampert, "Advanced optical materials for energy efficiency and solar conversion," *Sol. Wind Technol.*, vol. 4, no. 3, pp. 347–379, 1987, doi: https://doi.org/10.1016/0741-983X(87)90067-1.
[15] J. C. C. Fan, F. J. Bachner, G. H. Foley, and P. M. Zavracky, "Transparent heat-mirror films of TiO2/Ag/TiO2 for solar energy collection and radiation insulation," *Appl. Phys. Lett.*, vol. 25, no. 12, pp. 693–695, Dec. 1974, doi: 10.1063/1.1655364.
[16] S.A. Hassanzadeh-Tabrizi, "Precise calculation of crystallite size of nanomaterials: A review," *J. Alloys Compd.*, vol. 968, p. 171914, 2023, doi: https://doi.org/10.1016/j.jallcom.2023.171914.
[17] O. S. Heavens, "Optical properties of thin films," *Reports Prog. Phys.*, vol. 23, no. 1, p. 1, Jan. 1960, doi: 10.1088/0034-4885/23/1/301.
[18] "International Journal of Nanoscience and Nanotechnology." Accessed: Jul. 13, 2025. [Online]. Available: https://www.ijnnonline.net/