Pak. j. sci. ind. res. Ser. A: phys. sci. 2026 **69A**(2) 66-72 https://doi.org/10.52763/PJSIR.PHYS.SCI.69.2.2026.66.72

# A Comparative Study of the Optical Properties of ZnO: NiO NPs Synthesized by Plasma Jet and Pulsed Laser Ablation

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(received November 06, 2024; revised April 16, 2025; accepted April 21, 2025)

**Abstract.** This research aimed to investigate the optical characteristics of ZnO:NiO nanocomposites synthesized using two distinct methods; plasma jet to synthesize nanoparticles and pulsed laser deposition (PLD) for thin films formation. SEM micrographs demonstrated distinct samples structures; the films made with a pulsed laser had a more homogeneous and regular surface while the irregular and clustered nanoparticles produced by the plasma jet were observed. All of these differences had an impact on optical characteristics. Specifically, for the plasma jet technique, changing the post-reaction gas flow from 0.5 to 2.5 L/min decreased the energy gap from 5.09 eV to 4.52 eV; for the PLD technique, increasing the laser energy from 700 to 1000 mJ decreased the energy gap from 3.9 eV to 3.11 eV. The results highlight that the conditions of its fabrication play a significant role in tuning the electronic and optical properties of the material. Hence, ZnO:NiO can be regarded as a prospective material for optical and electronic applications.

**Keywords:** plasma jet, pulsed laser deposition, nanoparticles, optical properties, thin films, energy gap

## Introduction

Nanoparticles are extremely small measuring from 1 to 100 nanometers (Mazhir *et al*., 2021). Due to their unique properties in contrast with bulk materials, they have attracted a lot of interest lately (Abd El-Salam *et al*., 2024; Rashid *et al*., 2022). Although materials in the nano-scale are vital in various optical and electronic applications including sensors, medical engineering, and flexible displays primarily due to their enhanced light-interacting properties (Ahmed *et al*., 2024; Mohammed and Mohammed, 2023; Mohammed *et al*., 2022a; Youssef *et al*., 2019), Such materials are employed in applications due to their outstanding optical and electronic properties such as metal oxides that have nanosized characteristics such as zinc oxide (ZnO) and nickel oxide (NiO) due to enhanced surface area and reactivity (Issa and Aadim, 2024a and b). Nanoparticle synthesis can be achieved with one of several methods, where pulsed laser deposition (PLD) and plasma jet are most common. While PLD employs high-energy laser pulses that bombard a target to vaporize nanoparticles into a substrate, thereby facilitating the formation of fine nanomaterials (Issa and Aadim, 2024c; Shehab and Aadim, 2021; Jamal *et al*., 2020), the plasma jet relies on the formation of a dense plasma by a flowing gas, yielding unique nanoparticles with specific properties (Mohammed *et al*., 2022b). Though the synthesis of nanomaterials by both plasma jetting and pulsed laser deposition (PLD) is well explored, there is a lack of systematic comparative studies on their effect on the properties of nanosize ZnO:NiO composites. Some of the previous works proved the successful fabrication of several nanostructures based on these methods such as SnS:Ag doped Cu thin films (Almaula *et al*., 2022), silver nanoparticles (Mohamed *et al*., 2022a), magnesium oxide nanoparticles (Abbas and Aadim, 2023) and CuO/ZnO nanocomposite (Mohammed *et al*., 2023). Nevertheless, the role played by some important synthesis parameters, e.g. gas flow rate in plasma jetting, laser pulse energy in pulsed laser periodic deposition. The optical and structural properties of ZnO:NiO has not been satisfactorily addressed. Hence, this work studies the role of these parameters systematically to fill up the gaps and generate some utilization potential data on ZnO:NiO nanocomposites from the viewpoint of optical and electronic applications.

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## Materials and Methods

**Plasma jet method nanoparticle formation.** A cold-atmosphere plasma jet system consists of several key components that ensure optimal performance. The neon light transformer is used as a high-voltage power supply, in which the power output is 13 kV and 0.5 mA at a frequency of 50 Hz. This converter reduces overall system costs by replacing the traditional DC power supply, which is usually expensive. A voltage controller includes the task of adjusting the primary voltage of the high-voltage transformer, ensuring optimal performance of the jetting system. The power supply is designed to include overload, open circuit and ground leakage protection, enhancing system reliability and safety. The plasma jetting system comprises two parallel stainless-steel needles separated by a 2.75 mm thick Teflon insulator. The gas flow system ensures that gas is supplied at the correct flow rate to the plasma using a high-pressure gas storage cylinder containing argon gas. Flow rates range from 0.5 to 2.5 L/min, ensuring gas is used efficiently and effectively and Fig. 1 shows how it works.

**Pulsed laser deposition method for forming thin films.** The Nd-YAG laser system used operates at 1064 nm wavelength, with 100 to 1000 mJ pulse energy, 5-10 ns pulse width, 1-10 Hz frequency, 3-8 mm beam diameter and 1-5 J/cm² fluence depending on the settings. In this research, the deposition was completed utilizing a Nd-YAG laser with laser energies of (700-1000) mJ, peak duration of 9 ns, a repetition rate of 6 Hz and many pulses (200 shots). For deposition ZnO: NiO with x=0.5 concentrations on the glass substrate, the film was deposited at room temperature. The beat laser deposition is generally completed inside a vacuum chamber in $2.5\times10^{-2}$ mbar vacuum conditions. The arrangement of the substrate holders is targeted with the laser beam inside the chamber. The centered Nd: YAG SHG Q- exchanges a laser ray coming through a window, falling on the target's surface, creating an edge of 45°. The substrate is located in front of the target, with its surface parallel to the target. Adequate space is taken between the substrate and the target, so the substrate holder does not impede the falling laser ray. The work method is shown in Fig. 2.

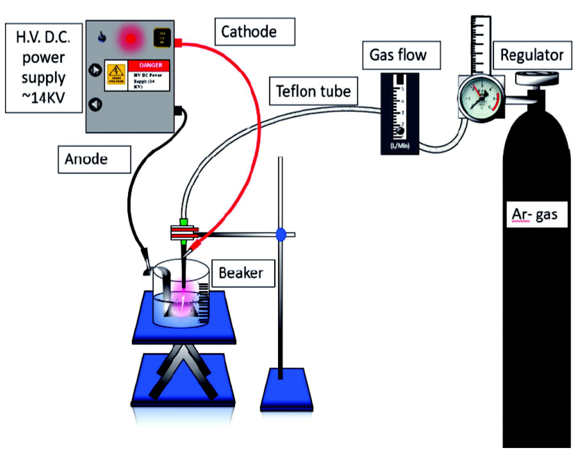


**Fig. 1.** The experimental setup of an atmospheric plasma jet with a high voltage D.C. power supply.

## Results and Discussion

The energy gap of nanoparticles prepared using plasma jetting is significantly affected by various factors associated with this manufacturing technique. By adjusting parameters such as plasma power, deposition time, gas flow velocity and substrate temperature, the energy gap of nanoparticles can be controlled. These parameters affect the kinetic energy and the plasma density, ultimately impacting the energy transferred to the material. In addition, the chemical composition of the gases used in the plasma jetting method can affect the energy gap. Results from optical absorption spectra measurements, conducted using Ultraviolet-visible spectroscopy of ZnO:NiO nanoparticles, as shown in Fig. 3, indicate that the absorption intensity increases. This increase in absorption intensity, particularly the observed red shift in the absorption edge, supports the narrowing of the optical band gap, consistent with quantum confinement effects or reduced carrier recombination due to structural rearrangement at higher flow rates. The measurements also showed that the energy gap (Eg) decreases with increasing plasma flow rate, with Eg adjusted from 5.09 to 4.52, as shown in Table 1. This result is consistent with (Hakkoum *et al*., 2022).

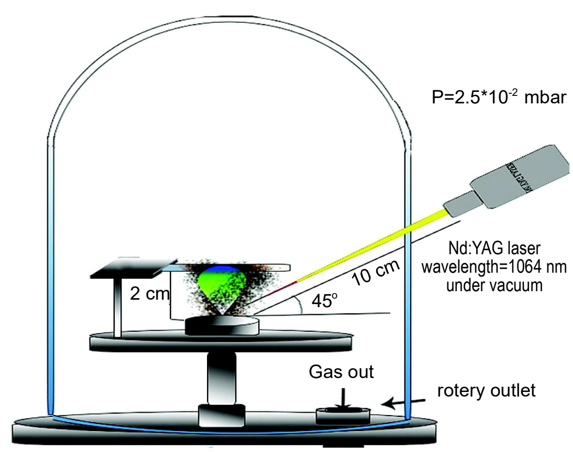


**Fig. 2.** Schematic of pulse laser deposition system.

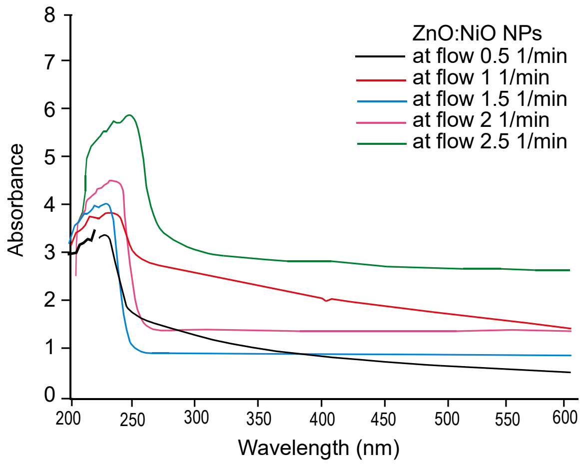


**Fig. 3.** Optical absorption spectrum of ZnO:NiO nanoparticles synthesized using the plasma jet method at different argon flow rates.

**Table 1.** Energy gap values of ZnO:NiO nanoparticles synthesized using the plasma jet method at different argon flow rates

| Sample code | Flow (L/min) | Energy gap (eV) |
|---|---|---|
| ZnO:NiO nanoparticles | 0.5 | 5.09 |
| | 1 | 4.7 |
| | 1.5 | 5.08 |
| | 2 | 4.88 |
| | 2.5 | 4.52 |

When plasma comes into contact with materials, plasma ions induce physical and chemical changes on the material's surface, such as etching and sputtering, which results in changes in the material's atomic arrangement and thus in its electronic structure. These changes may lead to the material's energy gap decrease, as shown in Fig. 4. This is consistent with (Aadim, 2016).

To determine the optical energy gap, researchers have used a relation that features the absorption coefficient, α; photon energy (hν) in electron volts (eV), a constant factor, known as $B_0$; and an optical energy gap in eV expressed $E_g$ opt, with an exponential coefficient (r) determining the type of transmission (Mohsin *et al.*, 2022):

$$\alpha h\nu = B_0 (h\nu - E_{g\,opt})^r \qquad (1)$$

As shown in Fig. 5, the optical absorption spectra of $ZnO_{(x)}$:$NiO_{(1-x)}$ thin films at x=0.5 reveal absorption peaks across the wavelength range from 200 nm to 1000 nm. As the laser pulse energy rises, the absorption of the thin films increases, consistent with previous studies (Mostafa *et al.*, 2021). The energy gap of thin films fabricated using the pulsed laser technique is significantly affected by several intrinsic factors associated with this technique. The pulsed laser method relies on the use of high-energy laser pulses to deposit thin films and the interaction between the laser pulses and the deposited material during deposition causes significant changes in the electronic structure of the

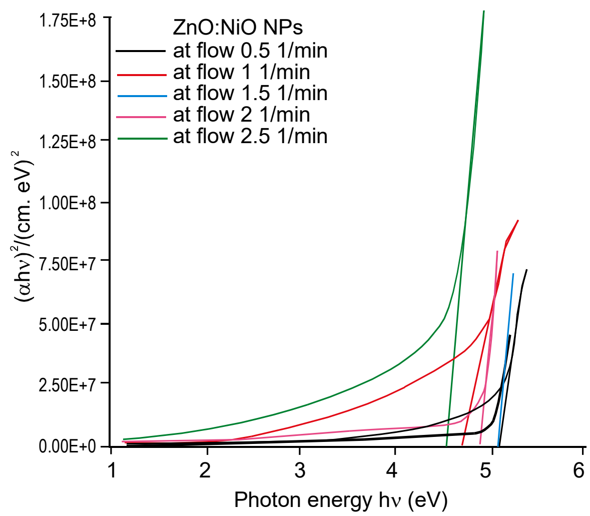


**Fig. 4.** $(\alpha h\nu)^2$ of the prepared ZnO:NiO nanoparticles synthesized using the plasma jet method at different argon flow rates.

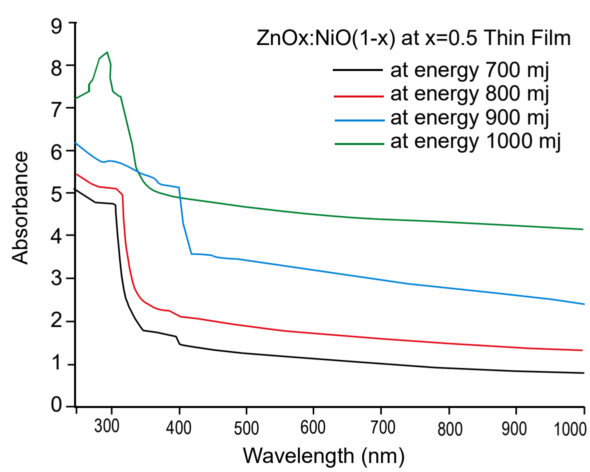


**Fig. 5.** Optical absorption spectrum of (ZnOx:NiO(1-x)) at x=0.5 thin films synthesized using pulse laser method at different energy.

material (Karim *et al*., 2024; Khudair *et al*., 2020). Important parameters affecting the energy gap of thin films include repetition rate, substrate temperature, laser pulse energy and pulse duration. According to studies, these parameters determine the kinetic energy and density of the laser-induced plasma, which influence the amount of energy delivered to the deposited material. The starting materials used also significantly affect the energy gap of thin films.

As shown in Fig. 6 and Table 2, the energy gap (Eg) of ZnO:NiO films reduces with rising laser pulse energy from 700 to 1000, with the energy gap being modified from 3.9 to 3.11. This is consistent with previous studies (Fahad *et al*., 2024). The effects of laser pulse energy on energy gap reduction depend on the specific material and the interaction mechanisms. When a material is exposed to laser pulses, photons can carry energy beyond the material's energy gap, exciting electrons from the valence band to the conduction band, thus reducing the required excitation energy. Intense pulses can induce

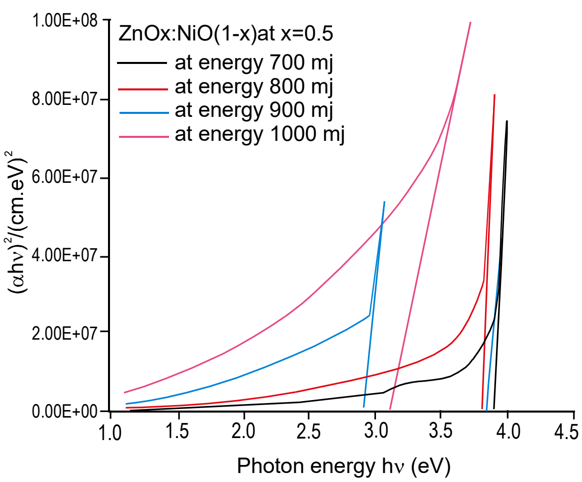


**Fig. 6.** $(\alpha h\nu)^2$ of the of (ZnOx:NiO(1-x)) at x=0.5 thin films synthesized using pulse laser method at different energy.

**Table 2.** Energy gap for (ZnOx:NiO(1-x)) at x=0.5 thin films synthesized using pulse laser method at different energy.

| Sample code | Energy (mJ) | Energy gap (eV) |
|---|---|---|
| ZnO: NiO thin film | 700 | 3.9 |
| | 800 | 3.8 |
| | 900 | 2.91 |
| | 1000 | 3.11 |

changes in the electronic structure, significantly reducing the energy gap. Furthermore, nonlinear optical effects such as multiphoton absorption or slip ionization can affect the energy gap, lowering the threshold required to excite electrons across the band gap.

SEM images (Fig. 7 and 8) show a clear difference in the nanoparticle structure between the thin films prepared using plasma jet and pulsed laser techniques, which is directly reflected in the measured energy gap values (Table 1 and 2). Analyzing the SEM image of the plasma jet sample (Fig. 7a), taken at 60.00 KX magnification

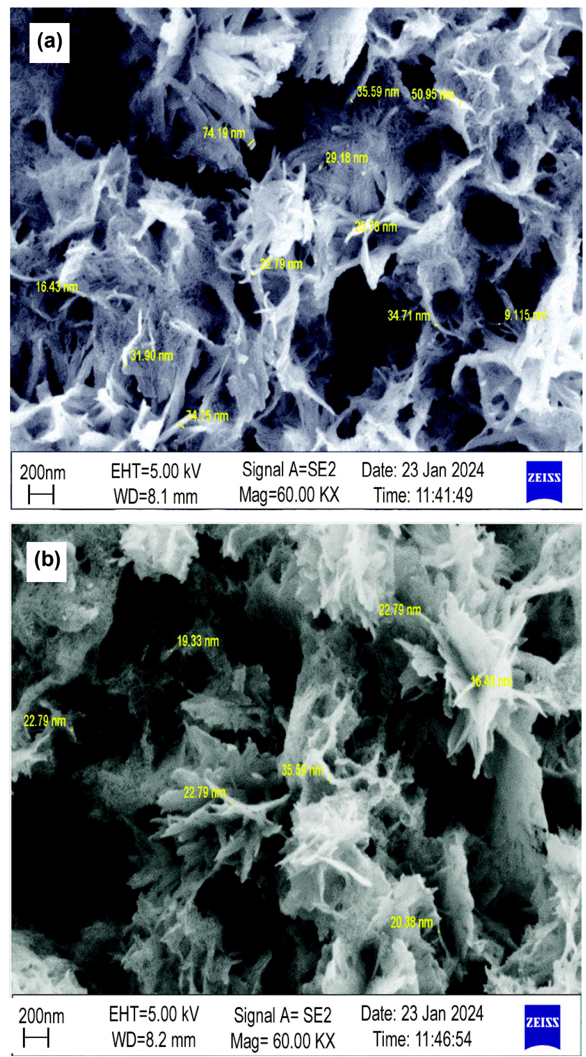


**Fig 7.** SEM image of the surface of the sample prepared by plasma jet technique, where the sample was prepared in case (a) at a gas flow of 0.5 L/min and in case (b) at a gas flow of 2.5 L/min.

with a scale bar of 200 nm, it can be seen that the particles are irregularly arranged, with varying sizes and some agglomerations, especially at the low flow rate of 0.5 L/min, where the energy gap was highest (5.09 eV). With the gas flow to 2.5 L/min, the SEM image (Fig. 7b) shows that the particles became larger and less agglomerated, reflected in a decrease in the energy gap to 4.52 eV. Interestingly, the energy gap does not decrease linearly with increasing flow rate. For example, after dropping to 4.70 eV at 1.0 L/min, the energy gap increased again to 5.08 eV at 1.5 L/min before continuing to decline. This suggests that factors such as transient defect states or temporary improvements in crystallinity might have influenced the band structure at intermediate flow rates, warranting further investigation. This can be explained by the effect of the increased gas flow rate on the rearrangement of the crystal structure and the reduction of optical defects in the material.

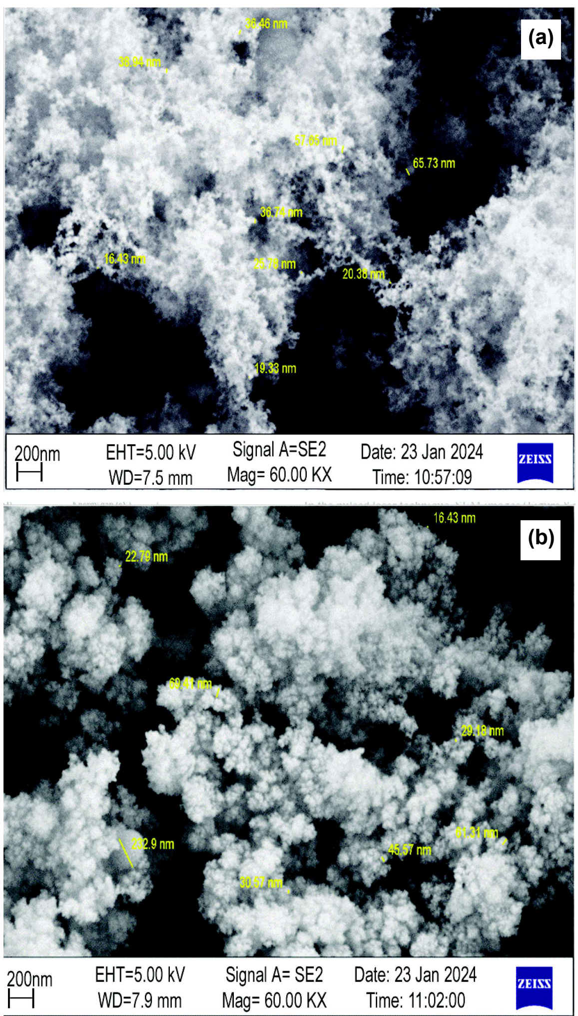


**Fig. 8.** SEM image of the surface of the sample prepared using laser, where the sample was prepared in case (**a**) with an energy of 800 mJ and in case (**b**) with an energy of 1000 mJ.

In the pulsed laser technique, SEM images (Fig. 8a and b) reveal a radical difference in the surface structure compared to the plasma jet technique. At 60.00 KX magnification with a 200 nm scale, it can be seen that the particles are more uniform and less agglomerated, contributing to the formation of more homogeneous films. However, the measured energy gap of the films prepared using this technique (Table 2) was lower, decreasing from 3.9 eV at 700 mJ energy to 3.11 eV at 1000 mJ energy. This can be explained by the fact that rising the laser energy causes recrystallization and a rise in the crystallite size, which reduces the energy gap. This is consistent with the SEM images, which show more distinct features of the homogeneous structure of the thin films compared to the nanoparticles formed by plasma jet.

To authenticate the recorded data for energy gap (Eg), the energy gap uncertainty for both the thin films and the nanoparticles were also identified. The nanoparticles made using plasma jet prepared had a standard deviation of ±0.25 eV; these values were consistent, with slight variations due to the particle size distribution. The higher standard deviation for the thin films prepared by pulsed laser technique was found out ±0.49 eV. These variations can be ascribed to several reasons such as differences in laser power, inhomogeneity in the deposition layer or modifications in the structure of the material during its growth.

## Conclusion

Absorbance and band gap analysis showed that, for plasma jet samples, the energy gap decreased from 5.09 eV to 4.52 eV as the gas flow rate increased. This trend suggests that changes in plasma conditions influence crystallinity and defect density. Meanwhile, for thin films prepared via pulsed laser deposition, the energy gap decreased from 3.9 eV to 3.11 eV with rising laser pulse energy, indicating modifications in the electronic structure due to increased crystallite size and surface homogeneity.

These findings underscore the importance of fabrication parameters such as gas flow rate and laser energy in tailoring the optical and electronic properties of ZnO:NiO materials. The ability to control band gap and particle structure through process optimization holds significant potential for optoelectronic and photocatalytic applications.

## Acknowledgement

The study was supported by the Plasma Physics Lab, Physics Department, College of Science, Baghdad University.

**Conflict of Interest.** The author declare that they have no conflict of Interest.

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