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ENHANCED SYNTHESIS OF NiO NANO FILM THROUGH SOL-GEL DIP COATING METHOD: INVESTIGATION THE IMPACT OF LASER IRRADIATION

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ABSTRACT:

The sol-gel synthesis technique was used to fabricate NiO nanofilms, and the process of dip coating aided by laser irradiation was used to form thin layers on silicon and glass bases at room temperature. By using lasers, a novelty was introduced to the technique that differs from another research. The thin layer was exposed to laser illumination (530 nm, 0.1, 1, 10 W) prior to heating during the deposition process. The main objective is to investigate the properties of thin layers further upon their formation as nanostructures using dip coating under different intensities of green laser light. We characterized the chemical bonding structure and surface morphology with the help of FTIR spectroscopy and SEM. Diffraction X-ray and UV spectroscopy were applied to evaluate the optical and structural characteristics of the films. Surface chemical composition was analyzed using EDS. The FTIR study demonstrated the existence of many exterior groups functional in the NiO nanofilms. NiO nanoparticles showed blue shifts in their infrared absorption band regarding the bulk NiO. The increase in NiOx film's optical bandgap was observed with the rise of laser irradiation intensity; values were found in the range of 3.5–4.01 eV. From the morphological analysis, the nanocrystalline grains in the films are uniformly covered. Through EDS testing, the composition of oxygen and nickel was confirmed. XRD measurements confirmed that during the synthesis process, cubic NiO nanoparticles were obtained. The effects of different power levels of laser irradiation on the films were discussed. According to the findings, High-irradiation laser in dip coating increased synthesis in the deposited films improved optical and structural characteristics adjusted for exact technological applications.

KEYWORDS: Sol gel technique, Nickel Oxide (NiO), Nano Film, Laser irradiation.

1. INTRODUCTION

Nanotechnology is one of the most active subfields in modern technique research. Nanoparticles have increasingly found applications because of their biological, physical, and chemical features. Because of quantum size effects, nanomaterials have attracted more attention because they are substantially smaller than bulk materials. Nanomaterials can exhibit some unique and impressive mechanical, electrical, magnetic, and optical properties that bulk materials cannot show. Nanomaterials from metal oxides, such as ZnO and NiO are currently said to be functional kinds of materials for technological applications, notably their unique optical, thermal, dielectric, vibrational, and characteristics of magnetization, along with some uses including catalytic activity, spin-polarized carrier sources, gaseous sensors, electronics, and others (Abbas *et al*., 2017; Guziewicz *et al*., 2011). Metal oxides show many different chemical and physical characteristics because they can adopt diverse structural geometries and demonstrate semiconductor, insulator, or even metallic characteristics within their electronic structure. Metal oxides are the primary category of functional materials that are essential to transmitting biological and chemical processes and sensing; besides, they were found to be great candidates for electrical and optoelectronic applications because of their distinct and adjustable physical properties. The ferromagnetic solid nickel oxide (NiO) has a Neel temperature of 523 K (Gandhi & Wu, 2017). It is a green colored crystalline substance. NiO is the focus of a sizable number of scientific publications due to its distinctive electrical, magnetic, and optical properties. An exceptional chemical stability has been seen in NiO band gap (3.6–4.0 eV) p-type semiconductors (Wang *et al*., 2015).

Due to its exceptional ion storage capabilities and low price, it is an intriguing subject of study. NiO nanostructures are a sort of p-type semiconductor that exhibit specific electric and magnetic characteristics that depend on the size of the particle. Nickel oxide nanoparticles are investigated as a functional material. Due to their specific mechanical, electrical, optical, magnetic, and thermal characteristics, these materials have great potential for use as electrochemical films, photoelectric devices, catalysts, battery electrodes, gas sensors, and other purposes (Abbas *et al., 2*017; Bi *et al*., 2004; Ichiyanagi *et al*., 2003). Several methods are used to produce NiO nanoparticles, like precipitation chemicals (Hotovy *et al*., 2001; Atul *et al.*, 2022); sputtering magnetron (Chen *et al*., 2021; Khoshhesab & Sarfaraz, 2010); chemical bath deposition (Onah *et al*, 2012); and sol-gel (Yan *et al.*, 2018; Zorkipli *et al.*, 2016). The use of the obtained nanoparticles depends on the selection of synthesis techniques (Mohsin *et al*., 2022; Zhao *et al*., 2018). In material science, the sol-gel process is a popular deposition technique for producing ceramic, optical, protective, and related surfaces. In order to progressively create a gel, this process begins with the hydrolysis of a liquid precursor (sol), then proceeds through poly-condensation. This gel is a biphasic system with a solid phase (integrated network, usually a polymer network) and a liquid phase (solvent). Step by step, the liquid's proportion decreases. To customize the solid's material qualities, the remaining liquid can be eliminated by drying and combined with a heat treatment. Compared to other wet chemical growth methods (CBD and spin-coating), dip-coating is considered to be one of the most suitable growth techniques for depositing large-area homogeneous, uniform, and compact coatings. Dip-coating has the advantage of

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having less precursor waste than spin-coating and the ability to reuse the sol (Faustini *et al*., 2010).

The NiO nanoparticles were grown on a glass and silicon substrate using the dipping technique. Before heating, the deposition was subjected to green laser irradiation. This study additionally looks into how the impact of the laser beam affects the surface morphology and characteristics of the structure and composition of the film synthesized. In contrast to the other dip coating method, we employed high-power laser irradiation to improve the dip coating during deposition. Just a single layer of coating was used until the extra effects produced by the laser were observed.

2. METHODS AND MATERIALS

NiO Synthesis

The chemicals, sourced from Sigma Aldrich, were used as received without further purification: tetrahydrate nickel acetate and monoethanolamide. A quantity of 0.122 g of nickel acetate tetrahydrate, containing 98% nickel, was combined with 50 mL of pure ethanol. The mixture was thoroughly mixed using a magnetic stirrer at 60 °C for a duration of 30 minutes. This process was carried out to produce nickel oxide using the sol-gel technique. A monoethanolamide complexing agent was used to prevent precipitation; the molar ratio of the ethanolamine to the Ni was maintained at 1:1. After the addition of MEA, the solution was magnetically stirred at a temperature of 60 °C for half an hour. The combination was a bright green color, as seen in Figure 1. After that, it was ultrasonically subjected at 40 °C for 15 minutes. After ageing the sol mixture for 24 hours, the prepared NiO sol solution was found to have a suitable film formation property, homogeneity, transparency, and no precipitation, as depicted in Figure 1.

Process of depositing nickel oxide nanoparticles as thin films

The deposition of NiO nanoparticles was conducted using the dip coating technique over both glass and silicon sheets that are wafers P-type (111) orientated with a resistivity of 1–30 Ω· cm and dimensions of 1.5 cm × 1.5 cm. The substrate was ultrasonically cleaned using deionized water and ethanol and then blown dry by a hot air blower before deposition. After being cleaned, the Si substrates and glass were dipped into the starting solution and removed vertically for 30 seconds. In the open air, with heat sources drying and modifying, the preparation took place based on dried samples. Thermal sources at 200 °C were used for 5 minutes, while green laser irradiation sources at varying power levels, a stable, consistent light output (continuous laser), and a distance of 5 cm from the target were used to achieve deposition on the film surface for the same duration as thermal sources at 200 °C, as detailed in Figure 1.

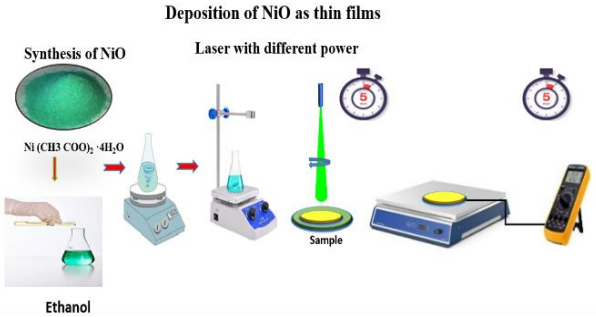

Figure 1: A schematic diagram synthesizing and depositing NiO nanoparticles as a thin film.

Characterization of Thin Film

Typical experimental methods are extensively used to characterize the nanoparticles. The chemical composition was determined by analyzing it using FTIR IRAffinity1S, which operates in the mid-infrared range of 400–4000 cm1. The UV visible spectroscopy investigation was verified on a double beam spectrophotometer (Hemadzu UV-1800 UV-vis) that operates across the range of 190–1100 nm. The exterior structure of the films was assessed by fast-emission electron microscopes with scanning (Cam Scan MV2300). The chemical structure of the film was analyzed by EDS together with SEM. The crystal structure of each sample was determined using a Rigaku C/max-2500 diffractometer with Cu Kα (λ = 1.5406 Å), which operated at 40 kV and 30 mA. The goal was achieved by a method called X-ray diffraction (XRD).

3. RESULTS AND DISCUSSION

Study of FTIR

The second figure displays the FTIR spectrum of nanoparticles of NiO subjected to different laser irradiation levels. The spectrum exhibits many major absorbing peaks, with the most prominent peak detected at 402.98 cm^{-1}. This peak corresponds to vibrations of the Ni-O bond in the FTIR spectra of NiO nanoparticles (Vijaya Kumar *et al.*, 2019; Ivanova, 2022., Khandagale & Shinde 2010); peaks at 671 cm^{-1} indicate having a Ni-O bond, as they correspond to the vibrations that stretch metal-oxygen bonds(Rahdar *et al*., 2015; Singh *et al.*, 2022). A broad absorbed band centered at 3519 cm1 with reduced peaks on both sides indicates the existence of multiple O-H groups in different environments, similar to those observed in polyols (Dehno Khalaji, 2013; Jawad *et al.*, 2021; Qiao *et al*., 2009; Ivanova, 2022). An additional sign at 1518.72 cm^{-1} indicates the existence of carbonyl (C=O) or dual (C=C) bonding in carboxylic acids or aromatic compounds (Singh *et al.*, 2022; Prabhu et al., 2022; Qiao *et al.*, 2009).

Previous peaks were frequently observable; they showed an increase in width and intensity as laser power was amplified. This is seen in P3, where broad band absorption within the 500–700 cm^{-1} range is associated with the stretch vibrating mode of Ni-O. The broad nature of this band shows that NiO molecules are in nanocrystals; the infrared peak corresponding to the stretching vibration of Ni-O bonds in NiO nanoparticles showed a blue shift. Due to the smaller size of the samples used in this study compared to the bulk forms of NiO, their spherical nanostructures, and the presence of a quantum size effect, NiO nanoparticles exhibit blue-shifted FTIR absorption in comparison to the bulk form.

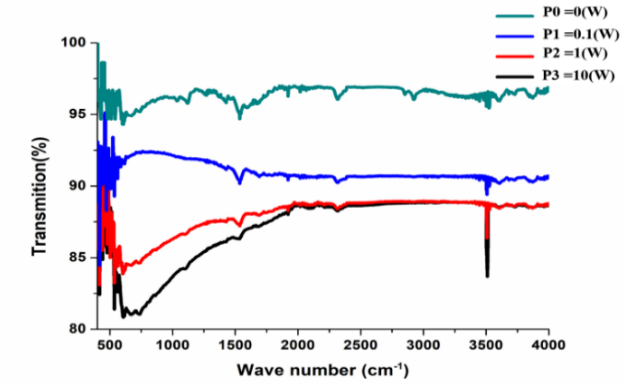

Figure 2: The FTIR spectrum of sol-gel nanoparticles made of nickel oxide films that were treatment with different power levels P 0 = 0 W, P1 = 0.1 W, P2 = 1 W, and P3 = 10 W.

Optical characteristics, Ultraviolet

Among the most significant methods for finding the energy arrangement and optical properties of nanocrystalline semiconductors is UV-V is absorption spectroscopy. The optical characteristics of NiO films using multiple laser powers were measured. Figure 3(a) displays the absorbance spectra of nickel oxide films for laser strengths ranging from 0 to 10 W, covering 200 to 800 nm. An absorbing edge is found within the range of 280–350 nm. The blue shift in the edge of absorptions may be related to several processes, including changes in quantum confinement and structural and exterior impacts of the nanoparticles (Rahdar *et al*., 2015). Furthermore, peak absorption wavelengths of NiO nanoparticles obtained by depositing are measured to be 280.5 nm. This measurement value differs from other samples due to differences in the size and deposition of the NiO nanoparticles, as seen in Figure 5 SEM pictures. Increasing the intensity of a laser results in a decrease in wavelength; decreasing the strength of the laser results in the emergence of absorbance at the absorption edge; and the absorption bands may have been redshifted as a result of a change in particle size (Hong *et al.*, 2021). Thus, the optical gap band energy of NiO nanoparticles can be determined using Tauc's method (Tauc, 2012, Pankove, 1975) , and some such measurements are seen in Fig. 3(b). The obtained values are 3.5 eV, 3.86 eV, 3.92 eV, and 4.01 eV, by using:

$$(\alpha h\nu)^n = A\,(h\nu - E_g)$$

The n value can be either ½ or 2 for an indirect and direct bandgap transition, respectively. The symbol hv denotes the light energy, α refers to coefficient absorption, and A is the proportionality constant. The formula demonstrates the extrapolation of the linear portion of the (αhv) n-hv curve to a zero hv value, which determines the optical band gap of the material. Table 1 shows the nanoparticle band gap energy for different powers of laser irradiation. The bandgap values increase with increasing laser irradiation power, possibly due to a reduction in the size of the crystals, which leads to quantum confinement effects (Esmaeel *et al*., 2024). The observed fluctuation in energy absorption shown by the NiO thin films is attributed to possible alterations in the film's structure over exposure to a powerful laser. Defects probably exist or vacancies are formed at the intergranular areas, which is responsible for the increasing trends of band gap energy when the particle size decreases. These newly formed states in the imperfections result in increase in bandgap energy. In Figure 4, a complete presentation of gap values as a function of laser power for NiO films is shown.

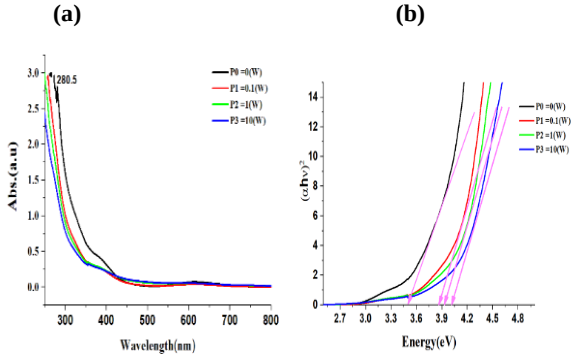

Figure 3 a: a graph showing the connection between the absorption (αt) and length wave (λ) of NiO nanofilm across various laser levels. b: (αhv)2 and energy for NiO thin films under different laser energies.

Table 1: The laser power and characteristics of NiO nanofilms, the absorbance peaks, and the optical band gap.

Sample	Power of Laser (W)	Absorbance peak	Energy gap (eV)	References
S_0	10	-	4.01	(Kumar and Basha, 2018)
S_1	1	-	3.92	(Mahmoud *et al*., 2011)
S_2	0.1	-	3.86	(Ghougali *et al*., 2016)
S_3	0	280.5	3.5	(El-Kemary *et al*., 2013)

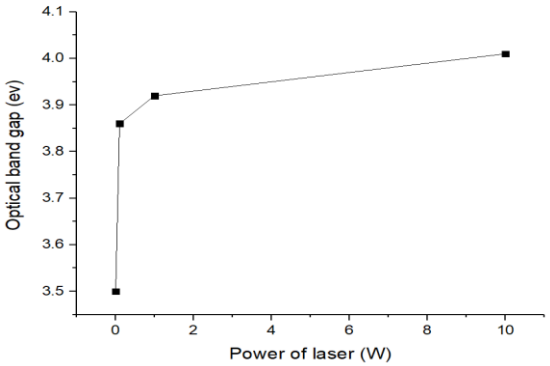

Figure 4: Estimated sol-gel NiO film optical band gap values based on green laser power.

Morphological properties, scanning electron microscopy

It is necessary to examine the outer morphology of the produced films since the surface morphologies of the samples affect their properties, which are crucial for sensing device applications. The surface morphological changes of the NiO synthesis films were carried out at different laser powers using a scanning electron microscope. The most often used method is scanning electron microscopy (SEM), which enables sufficiently high resolution to identify nanostructures. These methods are mostly used to examine the NPs' morphology and determine whether or not they are aggregated into supramolecular structures. Additionally, the particles's narrow and smaller size distributions within a particular range are visible. Morphology of NiO structure SEM published in literature with several techniques include (Kaliyaperumal *et al.*, 2022; Saghatforoush *et al*., 2012; Zorkipli *et al.*, 2016). Figures 5 a, b, and c are SEM exterior photo profiles obtained for the nanofilms processed at lesser powers (0, 0.1, and 1) prior to 200 °C of heating. The pictures indicate that the exterior of the film sample showed a nonuniform distribution of nanoparticles with the size of the grain increasing randomly. From Figure 5(d), on enhancing the laser power to 10 W, the size of the nanoparticle grains was found to decrease from being a sphere-like form to being similarly and equally dispersed. Here, morphology was better observed in previous literature, which was synthesized by dip coating (Sun *et al.*, 2017). Thus, one layer was used for the film's dip coating for a more evident effect of laser irradiation on the coating. It is, however, shown that the use of high power for coating films or

the absence of lasers makes a significant difference. A very smooth and uniform surface could be deposited in the case where laser irradiation was used in the film coating process to improve the surface morphology of the deposited material. It is evidently shown that laser exposure diminishes the grain size to a lesser value.

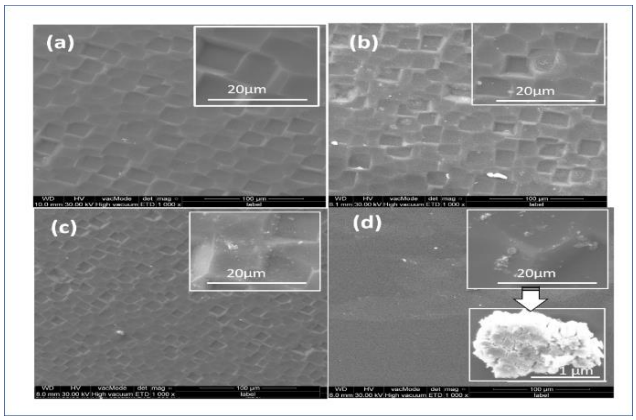

Figure 5: The SEM images of nickel oxide nanofilms processed at different power levels a = 0 W, b=0.1 W, c=1 W, and d=10 W.

EDS for elemental analysis

Figure 6 displays the findings of examining the composition elements of the components in the optimized NiO film using the EDS. The NiO samples free of contaminants had the maximum proportion of nickel and oxygen, as shown by the EDS spectra. In addition, the spectrum in Figure 6, which displays three distinct colors of the EDS (Si-a, Ni-b, O-c, and NiO-d), provides evidence supporting stoichiometry for the nanostructured NiO, which is being investigated. Examination of the XRD data was verified through EDS findings demonstrating the effective preparation of the NiO thin films.

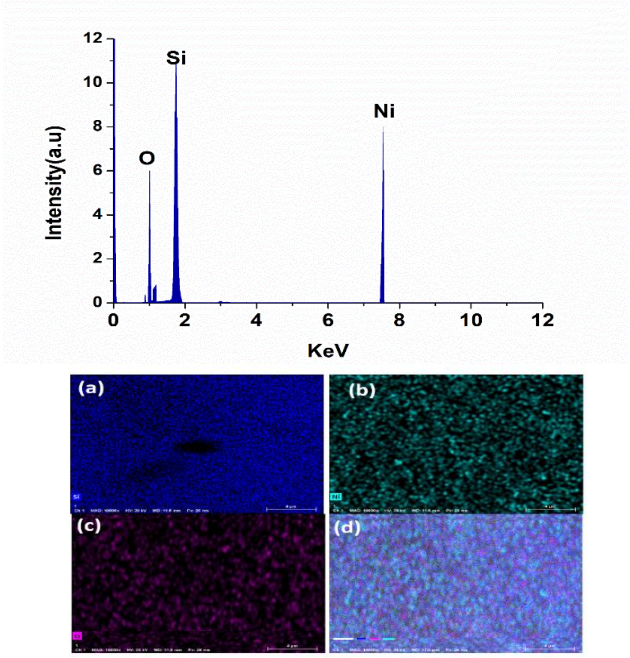

Figure 6: Displays EDS spectrum of P3=10(W) showing the presence of (Ni) and (O) components.

XRD for Structural Properties

It is irreplaceable to use X-ray diffraction (XRD) measurements to verify the produced NPs' purity. We used XRD analysis to examine the crystal compound of the NiO nanoparticle sample, labelled P3=10(W). The Bragg peaks were detected at three different angles: 38.33°, 44.58°, and 64.92°, as seen in Figure 7. X-ray diffraction peaks at 2θ of 44.58o were determined to be with miller indices of the hkl plane [200], another peak detected at 2θ of 38.33o was recognized as NiO [111], the XRD pattern revealed the formation of the cubic phase of NiO (JCPDS Card 47-1049), and the and the remaining peak discovered at an angle of 2θ of 64.92o was defined as Ni2O3, corresponding to the miller indices of the hkl plane [004] (Srivastava, 2010; Sharma *et al.*, 2015; Venkatachalapathy, 2022; Khalaji *et al.*, 2021). Hence, this result confirmed the formation of nickel oxide nanoparticles, while a small number of extra peaks showed contaminants.

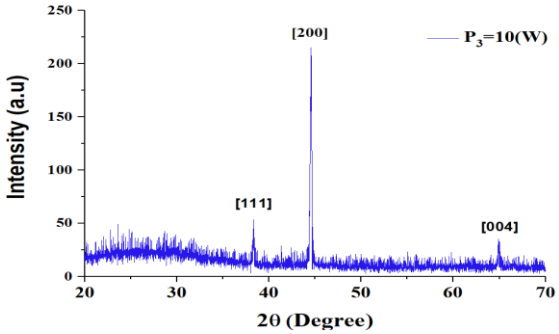

Figure 7: XRD peaks corresponding to the synthesized nickel oxide nanoparticle.

CONCLUSION

This work has demonstrated that the intensity of green laser light influences the synthesis of nanofilms and their structural and optical properties. Laser light (530 nm and power levels of 0.1, 1, and 10 W) was used in the preparation process. The presence of NiO nanoparticles and all functional groups was ascertained from FTIR analysis. UV–vis absorption spectra revealed that NiO NPs have the wavelength for absorption edge and their energy band gap is 280.5 nm and 3.5 eV, respectively. Utilizing powerful laser energy in the process of deposition increased the capability of the NiO films to absorb light in the visible range and increased their optical band gap by optical absorption spectra from 3.5–4.01 eV. SEM pictures indicated that as laser irradiation intensity increased, the grain size of the nanoparticles decreased, resulting in a uniform and homogeneous distribution with a spherical shape. XRD features have revealed the formation of the cubic structure of NiO nanoparticles. The successful synthesis of NiO nanofilms by assisted high-power green laser irradiation was confirmed by EDS and XRD analysis.

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DECLARATION

The author of this work confirms that there have not been any conflicting interests.

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