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Tailored Synthesis of NiO Thin Films through Spray Pyrolysis

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ABSTRACT

In this study, a NiO thin film was prepared onto a glass slide by spray pyrolysis technique. The spray pyrolysis method finds widespread usage for it being versatile, low-priced, and yielding thin films of very good quality. Expectingly, high resolution XRD was used to study the structural properties of films, and FESEM was utilized for surface feature studies. The study revealed that the film has a cubic crystal structure. The surface was smooth, dense, and uniform, and the FESEM images showed tiny flower-shaped grains.

Keywords: Nickel Oxide, Spray Pyrolysis, Thin Film, XRD, FESEM, Cubic Structure

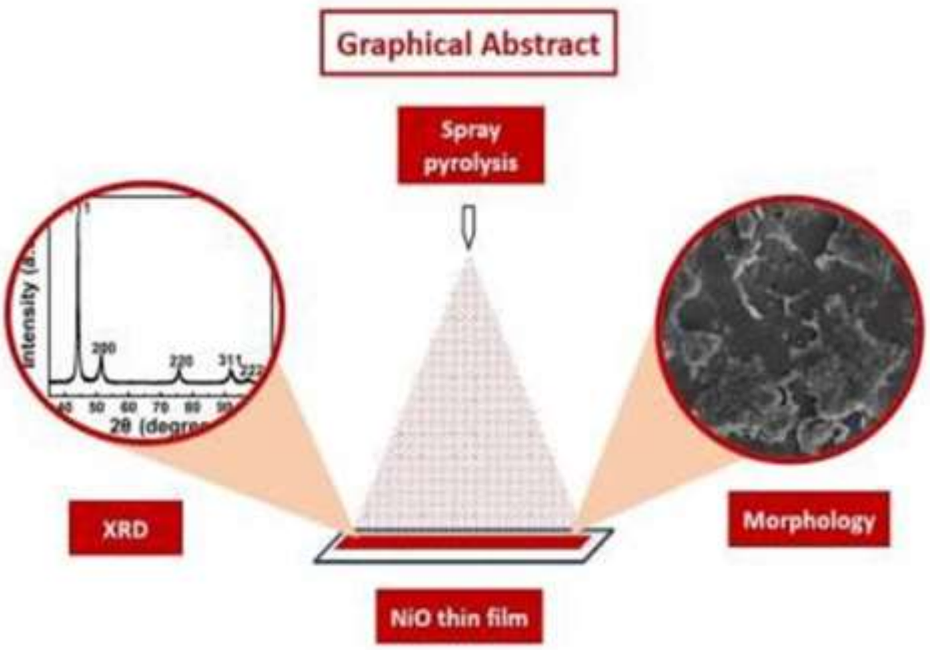

Fig1: Graphical Abstract

1. Introduction

Thin films, with their unique, tunable properties, have attracted the attention of scientific researchers and industries alike. There are thousands of applications that thin films could find use in, such as sensors, optoelectronic devices, detectors, and energy storage. “Nickel oxide (NiO), being a binary transition metal oxide, is a well-known p-type" and hence, has certain properties with a relatively low band gap, chemical stability, and electrical behavior. It is due to these characteristics of NiO that applications in gas sensing, supercapacitors, photocatalysis, and solar energy devices stand on promising grounds.

The various deposition procedures that attract attention for depositing uniform NiO thin films with more adherences include “chemical vapor deposition, sputtering, pulsed laser deposition, electrochemical deposition, chemical bath deposition, SILAR, and spray pyrolysis. Spray pyrolysis finds preference mainly because it is simple and low-cost and can be used for large-area coatings also”. Since this technique works in atmospheric conditions, it neither requires the use of dielectric materials for substrates nor mandates the equipment of high standard afforded by high-vacuum coating processes. Hence it is validated on an industrial scale, various aspects making this spray pyrolysis technique quite auspicious for commercial production.

"NiO thin films were deposited through spray pyrolysis in this work. Structural and surface characterization on the deposited films was carried out, respectively, by X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM)".

2. Experimental Details

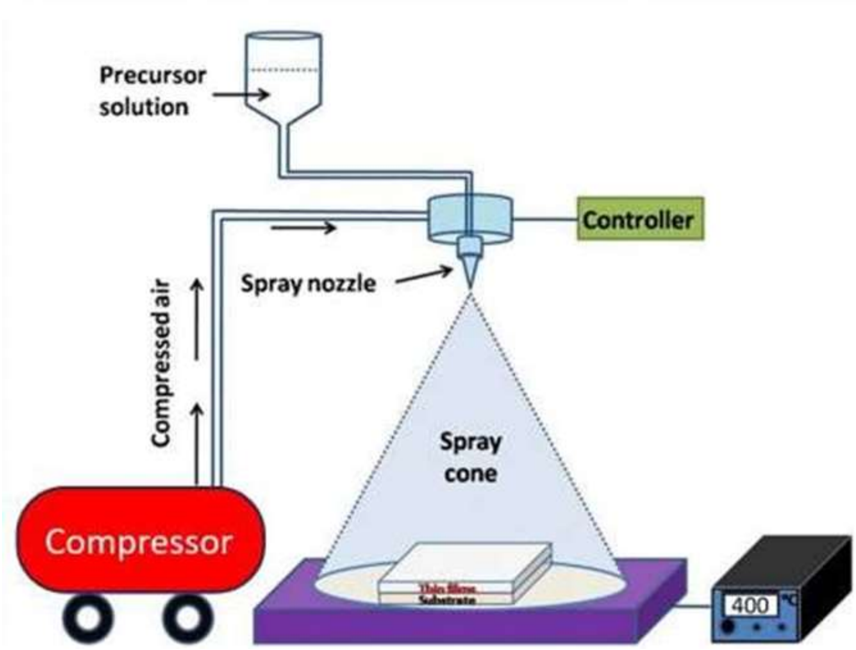

***Fig**2: Spray Setup*

Proper cleaning of the substrate is a limiting step in depositing high-quality thin films endowed with good uniformity and high adhesion strength. In the present investigation, soda lime glass substrates (with dimensions of 76 × 25× 1 mm) were employed for depositing NiO thin films. In the beginning, these glass slides were washed several times with a weak soap solution to get rid of surface contaminants. Afterward, the slides were immersed in chromic acid for 24 hours for thorough cleaning. Finally, they were rinsed thoroughly with distilled water to remove any residual chemical deposits.

Nickel nitrate was chosen as the precursor for these NiO thin films. A 1 M solution was prepared by dissolving the precursor in 20 ml of double-distilled water. "The beaker containing the precursor solution was placed on the magnetic stirrer, and stirring took place for 30 minutes to ensure complete mixing of the solutions before charging the spray gun. The film deposition was carried out by spray pyrolysis at a substrate temperature of 400 °C. Atmospheric gas was compressed and used as the carrier gas, with an ultimate flow rate of 20 liters/min. The distance between the nozzle and the substrate was maintained at 20 cm. The solution was sprayed at a rate of 2 ml/min for a time interval of 10 minutes".

After deposition, the NiO thin film was allowed to cool gradually to room temperature on the hot plate. The resulting films were then subjected to various characterization techniques to analyses their structural and surface properties.

3. Results and discussion

3.1 Structural Analysis

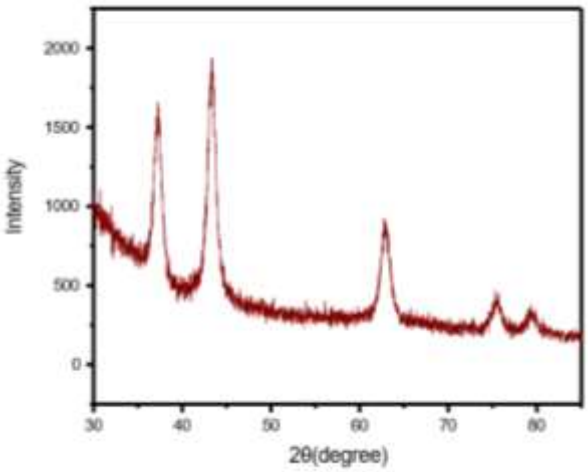

Fig3: XRD spectra of NiO and annealed NiO thin films.

"X-ray diffraction (XRD) is a widely used technique to investigate the structural characteristics of synthesized materials, particularly in powder or thin film forms. The XRD

pattern of the NiO thin film", as shown in the figure above, reveals multiple Bragg diffraction peaks, indicating the formation of a crystalline structure. These peaks confirm the presence of a face-centered cubic (FCC) phase in the as-deposited NiO film. Prominent diffraction peaks were observed at 2θ values of relevant angles for NiO, 37.2° and 43.3°, while several other low-intensity peaks were also detected. The experimental XRD data closely matches the standard reference pattern provided by JCPDS card No. 47-1049, validating the formation of single-phase NiO.

The identified diffraction planes correspond to various crystallographic orientations, such as the (111) and (200) planes. It is observed that higher deposition temperatures tend to enhance

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

both the number and intensity of diffraction peaks, indicating improved crystallinity and the emergence of additional growth orientations [12,13]. "To estimate the crystallite size (D) of the NiO thin film, the Debye-Scherrer formula was employed:

Here, β represents the full width at half maximum (FWHM) of the diffraction peak, k=0.9 is the shape factor, and λ=1.54 Å is the wavelength of Cu Kα radiation. Based on this analysis, the average crystallite size of the as-deposited NiO thin film was found to be approximately

10.5 nm".

3.2 Morphological analysis

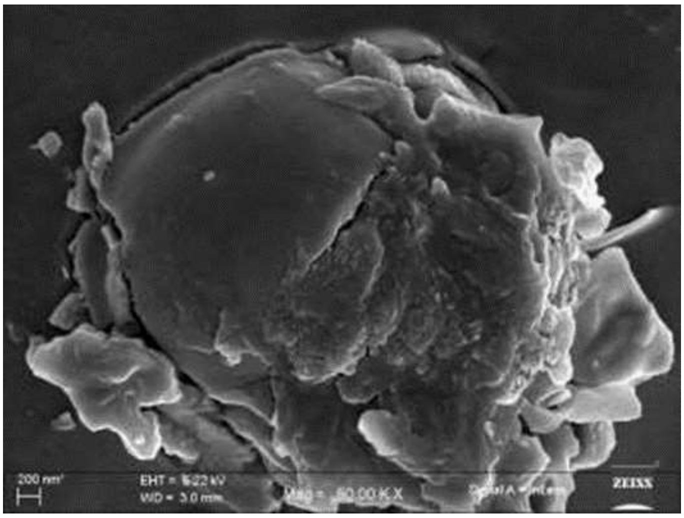

Fig4: FE-SEM images of NiO

The surface morphology of the NiO thin film revealed a uniform and compact arrangement of fine, capsule-shaped nanoparticles with minimal void spaces. These NiO nanostructures tended to aggregate, resulting in the formation of spherical clusters due to particle agglomeration**.**

4. Conclusion

The NiO thin film was fabricated "using the spray pyrolysis technique" onto a preheated substrate. The resulting film exhibited a dark, nearly black appearance and was found to be uniform, smooth, and well-adhered to the substrate surface. Structural analysis revealed that the as-deposited film crystallized in a well-defined monoclinic phase. The estimated grain size confirmed the formation of nanoparticles within the nanometer scale, indicating successful growth of nanostructured NiO.

These nanoparticles showed strong preferential orientation along the (-111) and (111) crystallographic planes. "Field Emission Scanning Electron Microscopy (FESEM)" images displayed a surface densely populated with clustered NiO nanoparticles. The surface was largely covered with capsule-like formations, with very few visible voids, suggesting a compact and continuous film morphology. Given these structural and morphological features, the as-prepared NiO thin film demonstrates promising potential for use in optoelectronic devices such as photosensors and solar energy conversion systems.

Credit authorship contribution statement

Abhilash: Central theme, Conceptualization, literature survey, Methodology, Graphical abstract, Data curation, Characterization analysis, Draft preparation. **Digvijay :** Methodology. **Chandrakant:** Supervision, Reviewing the manuscript.

Declaration of competing interest

"The authors declare that they have no financial or personal ties that could have influenced the outcomes or interpretations of the research presented in this study".

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