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# The Effect of Sputtering Time and Substrate Type on the Structure of Zinc Nanoparticles Prepared by the DC Sputtering Technique

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## HIGHLIGHTS

- The samples have a hexagonal wurtzite structure.
- All the samples showed a uniform distribution of granular surface shape morphology.
- Zn thin film thicknesses were increased as the sputtering time increased for all substrates.
- The best result was the deposition of zinc nanoparticles on Si (p-type) at 1 min, where the particle size was at the peak of 7 nm.

## ARTICLE INFO

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**Keywords:** Metal NPs/Si (111); DC sputtering method; FESEM;XRD diffraction.

## ABSTRACT

Zn thin films have been successfully deposited on two different substrates, FTO and p-type Si (111), with thickness (112, 186) nm at (1 and 8) min, respectively, via DC sputtering technique in this work. Structural properties of the prepared thin films were studied using X-ray diffraction (XRD) and field-emission scanning electron microscopy (FESEM). XRD results showed that the samples have a hexagonal wurtzite structure. From the results of FESEM images, all the samples showed a uniform distribution of granular surface shape morphology. The grain sizes of the Zn thin films were estimated based on measured X-ray diffraction patterns. Zn thin film thicknesses were increased as the sputtering time increased for all substrates. The best result was the deposition of zinc nanoparticles on Si (p-type) at 1 min, where the particle size was at the peak of 7 nm.

## 1. Introduction

The characterization of nanomaterials (1–100 nm) differs from the same material in bulk [1]. The physical and chemical properties and the ratio of surface to volume are completely changed in nano-size materials [2]. According to nanotechnology field development, wide types of nanoparticles (NPs) can be obtained that possess spatial characterizations, given a broad range of applications and scientific research fields [3]. For example, zinc nanoparticles can be classified due to their chemical structure: metallic nanoparticles (ZnNPs), nanoparticles of metal oxides (ZnONPs), and semiconductor nanoparticles (ZnS NPs, ZnSe NPs) [4]. The suitable method used for the preparation of nanoparticles can be chosen according to the application of nanoparticles. Many preparation methods could be utilized for getting nanoparticles with controlling structure and size. Although all the methods can obtain good NPs, they still need to develop the procedure, which can be easy in the industrial and commercial applications to obtain better production and more efficient yield [5-8]. Many techniques can be utilized for the deposition of Zn thin films on different substrates, such as the Hydrothermal Method [9], chemical vapor deposition [10], sol-gel method [11], pulsed laser deposition [12], radiofrequency sputtering, and DC sputtering technique. The DC sputtering technique is a simple and low cost to obtain metal and alloy thin films compared to other techniques [13]. The present work aims to investigate the structural properties and surface morphology of Zn thin films deposited on two different substrates (FTO coated glass and Si p-type) at two different times. The change in the structure was investigated using X-ray diffraction (XRD). The surface morphology of the thin films was also studied by scanning electron microscopy (SEM).

## 2. The experimental work

DC sputtering schematic diagram is shown in Figure (1). In the beginning, FTO coated glass and p-type Si (111) with 5–40 Ω cm resistivity, and 0.45 mm thickness were used as substrates for the deposition. These substrates with sizes of 1 cm x 1cm



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were cleaned with ethanol in a digital ultrasonic cleaner device (model / CD-4820) for 15 minutes. Then, the substrates were rinsed with distilled water, dried at room temperature, and placed on the anode inside the deposition chamber. A circular Zinc target with 4.7 cm diameter and 5 mm thickness with 99.9% purity supported by SIGMA-ALDRICH has been located on the cathode in the sputtering chamber. Argon gas with a maximum pressure of 1.5 bar and flow rate of 500 sccm was used to generate the plasma in the deposition chamber by glow discharge with a discharge current was 15 mA, and a rotary pump was used to discharge the tube to a vacuum below the $10^{-1}$ Torr. The anode was mounted approximately 7 cm above the cathode. In this work, we employed the sputtering time of 1 and 8 min to obtain different Zn thin film thicknesses, while 30 minutes was used to investigate XRD patterns. Zn thin film thicknesses were measured using the optical interferometer technique. The structural properties of the prepared thin films were examined by a (Phillips- Xpert) x-ray diffractometer with (λ=1.54056Å) and a (MIRA3 TESCAN) field emission scanning electron microscopy (FESEM).

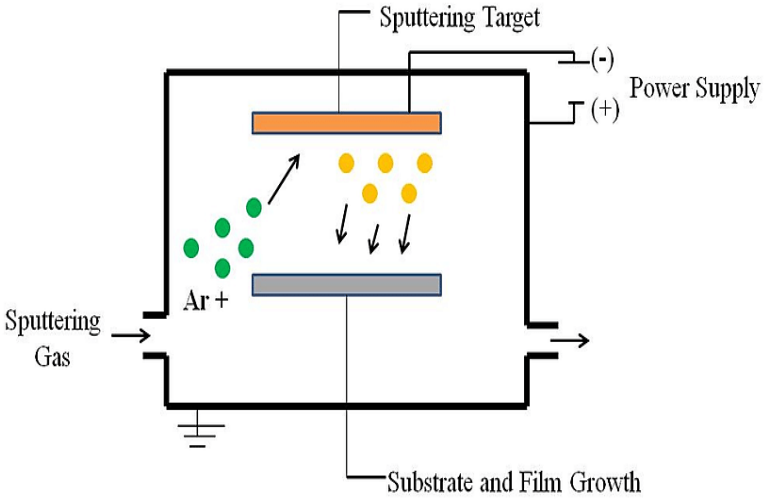


**Figure 1:** Schematic diagram of DC sputtering

## 3. Results and Discussion

An optical interferometer technique has been utilized to obtain the thickness measurements of the prepared thin films. This technique is based on the interference of light beams reflected from the thin film surface and substrate bottom. The diode laser of wavelength (532 nm) was used, and the thickness was determined using the equation:

$$d = \frac{\Delta x}{x} \times \frac{\lambda}{2} \quad (1)$$

Where *x*: Fringe width
Δ*x*: the distance between two fringes
λ: Wavelength of laser light.

X-ray diffraction patterns are shown in Figure (2) at the range of 2θ ≈ 10° to 80° for the Zinc thin films obtained by DC sputtering on FTO coated glass and p-type Si substrates. The peaks of XRD for Zn thin films on FTO coated glass substrate was shown at 2θ ~ 37° and 52° on the (1 0 0) and (1 0 2) plane, respectively, while peaks at the angles 2θ ~ 36°and 75° were seen on the (0 0 2) and (0 0 4) planes respectively, return to for Zn thin films on p-type Si substrate according to standards (JCPDS 36-1451). It shows the polycrystalline nature with the hexagonal wurtzite structure of the prepared thin films [14]. From figure (2), we can indicate that the prepared thin films on p-type Si showed high peak intensity over the plane (0 0 2). The (0 0 2) is the preferential plane for thin-film growth on Si substrate. Broad peaks at (1 0 0) and (1 0 2) plane on FTO and (0 0 2) plane on Si p-type are seen in this figure. These broad peaks arise due to the low crystallinity of the material. The effect of the substrate type on XRD patterns of Zinc thin films was clearly shown in Figure (2). The grain size and dislocation density were changed due to the substrate type. The growth of thin films on Si p-type exhibited greater grain size than that deposited on FTO coated glass substrate. Scherrer formula was used to determine the grain size of the zinc thin film (D).

$$D = K.\frac{\lambda}{\beta.cos\theta} \quad (2)$$

Where K is the shape factor of the average grain (0.9), λ is the X-ray wavelength (0.154056 nm for Cu Kα1), β is the full width at half maximum of the diffraction peak in radians, θ is the diffraction peak angle. From equation (2), the FWHM of the diffraction peak has an inverse relation to the grain size of the nanoparticles. As the nanoparticle grain size increased, the diffraction peak became sharper. The dislocation density (δ), which represents the length of the dislocation per unit volume of the crystal, can be determined from formula (3):

$$\delta = 1/D^2 \quad (3)$$

Thin-film parameters from XRD results are presented in Table 1. The surface morphology of the prepared films was identified using FE-SEM. Figure (3) shows a similar, isolated granular surface shape morphology with uniform distribution of ZnNPs prepared in this work. These nanoparticles were homogeneously distributed on the substrate surfaces. These images confirmed that, in a specific area, the distribution of granules that form the crystals was increased with the increase of sputtering time. For the prepared samples in 8 min, fewer FTO coated glass, and Si surfaces can be observed. Figure (4) shows the EDX mode of the FESEM analysis of zinc nanostructures synthesized on different substrates at different sputtering times. The EDX spectrum of each element composition was almost similar, except the weight of the elements was different due to the change in the sputtering time. For ZnNPs deposited on FTO coated glass substrate, approximately 11.2% and 21.4% weight of Zinc was present in the prepared nanoparticles for 1 and 8 min, respectively, while for ZnNPs deposited on p-type Si substrate, approximately 30.5% and 38.4% weight. The other percentages were for other elements, as shown in figure (4). The histogram of Zn nanoparticles on FTO at 1 min; figure 5 (a), changes from 3.5 - 35 nm, with a peak at 10.5 nm when deposition time rose to 8 min; figure 5 (b), the Zn nanoparticles varied between 8 - 80 nm with a peak of 32 nm. While the histogram of Zn nanoparticles on Si (p-type) at 1 min; figure 5 (c) results in nanoparticles dimensions varying between 1 - 10 nm, with a peak at 7 nm. Increasing the deposition time to 8 min, figure 5 (d), the Zn nanoparticles will be varied between 4 - 40 nm with a peak at 20 nm.

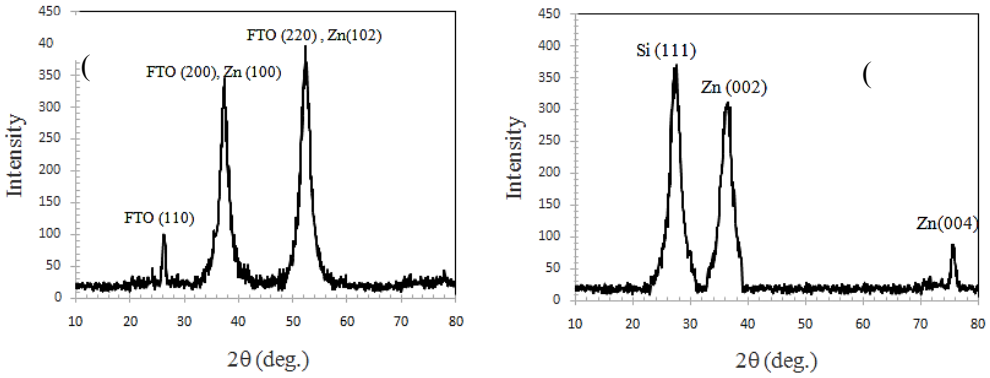


**Figure 2:** XRD patterns of Zn NPs on (a) FTO coated glass and b) Si (p-type)

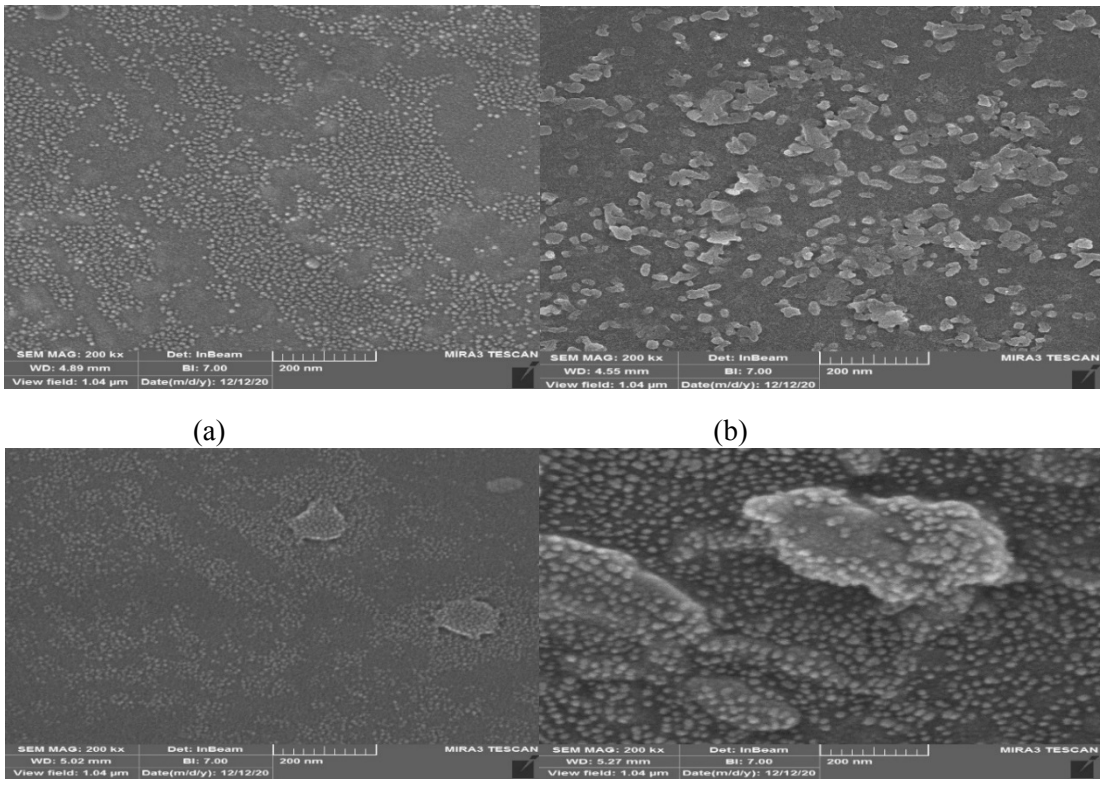


**Figure 3:** SEM images of Zn NPs on (a), (b) FTO and (c), (d) Si p-type at 1 & 8 min respectively

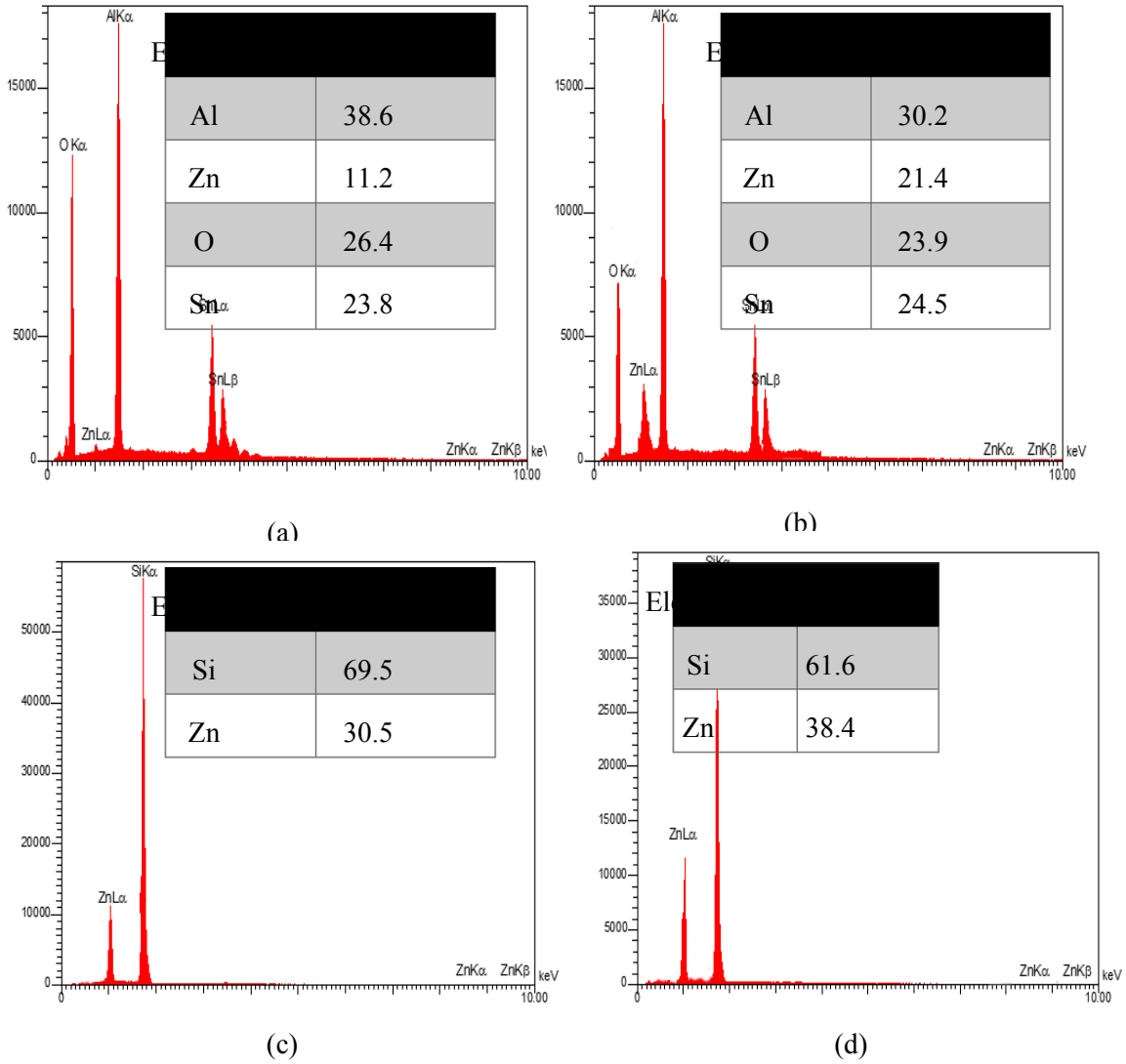


**Figure 4:** EDX at 1&8 min of (a), (b) Zn NPs on FTO coated glass and (c), (d) Zn NPs on Si(p-type)

**Table 1:** XRD parameter of the prepared Zinc thin films on FTO coated glass and Si (p-type)

| Substrate type | (h k l) | 2θ(degree) | FWHM(rad) | D(nm) =0.9λ/βcosθ | δ=1/D²(cm⁻²) |
|---|---|---|---|---|---|
| FTO coated glass | (100) | 37.325 | 0.039433 | 3.7 | 7.26E+12 |
| | (102) | 52.225 | 0.041877 | 3.7 | 7.36E+12 |
| Si (p-type) | (002) | 35.925 | 0.042285 | 3.4 | 8.42E+12 |
| | (004) | 75.375 | 0.010556 | 16.6 | 3.63E+11 |

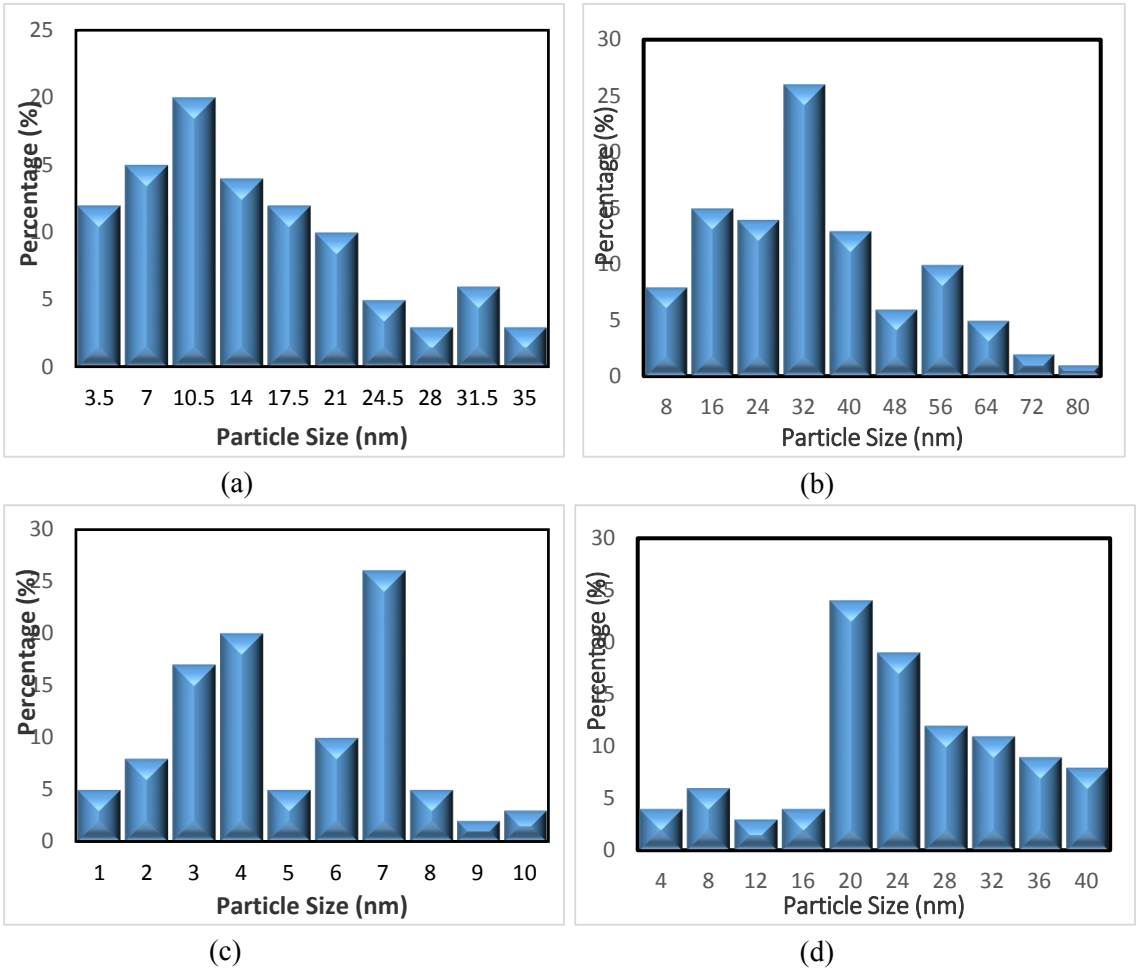


**Figure 5:** Histogram of ZnNPs size at 1&8 min of (a), (b) Zn NPs on FTO coated glass and (c), (d) ZnNPs on Si (p-type)

## 4. Conclusion

In conclusion, there is a feasibility to prepare Zn nano-thin films using the dc diode sputtering method. The influence of substrate type and sputtering time was studied. The thickness of the prepared Zn thin film was increased with increased sputtering time. The XRD results showed that the growth of hexagonal wurtzite structure had been successfully obtained for zinc thin films on FTO coated glass and p-Si (111) substrates via DC sputtering method. The grain size of Zn NPs deposited on p-type Si substrate was higher than that deposited on FTO. The surface morphology of the prepared Zinc thin films indicated the formation of a granular surface when Zn is deposited on FTO and p-type Si (111) substrate.

### Author contribution

All authors contributed equally to this work.

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### Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

### Conflicts of interest

The authors declare that there is no conflict of interest.

## References

**[1]** G. K. Salman, Effect of Zn substation on structural and magnetic properties of Cu ferrites nanoparticles, Eng. Technol. J., 34 (2016) 882-893.

**[2]** K. Ibrahim, S. Khalid, K. Idrees Nanoparticles: properties, applications and toxicities, Arab. J. Chem., 12 (2019) 908-931. https://doi.org/10.1016/j.arabjc.2017.05.011

**[3]** I. M. Ibrahim, Sh. I. Sharhan, F. T. Ibrahim, Hydrogen gas sensor of ZnO doping with CuO/PS nanocomposite, Eng. Technol. J., 33 (2015) 1093-1101.

**[4]** A. Attarad, P. Abdul-Rehman, Z. Muhammad, Elemental zinc to zinc nanoparticles: is ZnO NPs crucial for life? Synthesis, toxicological, and environmental concerns, Nanotechnol. Rev., 7 (2018) 413–441. https://doi.org/10.1515/ntrev-2018-0067

**[5]** K. Kannan, M. Manikandan ,Selection of a suitable method for the preparation of polymeric nanoparticles: Multi-criteria decision making approach, Adv. Pharm. Bull., 5 (2015) 57–67. https://doi.org/10.5681/apb.2015.008

**[6]** A. M. El Shafey, Green synthesis of metal and metal oxide nanoparticles from plant leaf extracts and their applications:A review , Green Process. Synth., 9 (2020) 304–339. https://doi.org/10.1515/gps-2020-0031

**[7]** F. Lin, W. Wei, A Combined Method of r-NPS and t-NPS evaluations for identification of negative triggers of detractors' experience, Sustainability, 12 (2020) 1652. https://doi.org/10.3390/su12041652

**[8]** A. A. Salman, W. K. Khalef, Characterization and photodetector of zinc oxide/Si thin films prepared by spray pyrolysis technique, J .Phys .Conf. Ser.,1795 (2021) 012051. https://dx.doi.org/10.1088/1742-6596/1795/1/012051

**[9]** Kh. S. Shaker, S. S. Shaker ,Preparation of ZnO like–nanoflower by hydrothermal method, Eng. Technol. J., 35 (2017) 178-181.

**[10]** N.Hacini, M. Ghamnia, M. A.Dahamni, A.Boukhachem, J-J.Pireaux, L. Houssiau, Compositional, structural, morphological, and optical properties of ZnO thin films prepared by PECVD technique, Coatings, 11 (2021) 202. https://doi.org/10.3390/coatings11020202

**[11]** S. M. Al-Jawad, M. M. Ismail, S. Emad, Effect of hydrothermal reaction temperatures on structural and optical properties of ZnS nanoparticles, Eng. Technol. J., 34 (2016) 808-819.

**[12]** S. S. Shaker, A. H. Mohammed, M. S. Khalaf ,Effect of laser pulses on characterization of zinc oxide thin film prepared by PLD, Eng. Technol. J., 36 (2018) 93-97. http://dx.doi.org/10.30684/etj.36.1B.15

**[13]** U. M. Nayef, M. J. Jasim, Characteristics study of ZnO thin films by rapid thermal oxidation treatment technique, Eng. Technol. J., 33 (2015) 37-44.

**[14]** B. N. Joshi, H. Yoon, H.Y. Kim, J. H. Oh, T. Y. Seong, S. C. James, S.S. Yoon,Effect of zinc acetate concentration on structural, optical and electrical properties of ZnO thin films deposited by electrostatic spray on ITO substrate, J. Electrochem. Soc., 159 (2012) 716-721. http://dx.doi.org/10.1149/2.077208jes