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Synthesis and Characterization of Microwave-assisted ZnO Nanostructures

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Abstract: ZnO nanostructures have been successfully prepared by microwave irradiation method. The synthesized samples were characterized by X-ray Diffraction method (XRD), Scanning electron microscope (SEM), Transmission Electron microscopy (TEM), and Energy Dispersive X-ray Analysis (EDAX) and Fourier Transform Infrared Spectroscopy (FTIR) technique. The absorption edge and band gap of ZnO were calculated by UV- visible spectra. Highly crystalline ZnO nanostructures have been obtained by the microwave assisted procedure. Furthermore, the formation of these ZnO nanostructures can be obtained within short times (5, 10 and 15 minutes) by a fine tuning of the microwave irradiation.
Keywords: Gas sensors, precursors, Zinc oxide, nanostructures, band gap and optical properties.

1. Introduction

In recent years, nanoscience and technology have potential applications in the field of science and technology. Particulary nanocrystalline materials are treated as the suitable material for those applications. Intensive investigations were carried out for most of the applications of these new classes of materials. Among the various nanocrystalline materials, ZnO with particle size in the range of several nm are treated as exclusively suitable material for various applications because of their unique properties [1]. Generally ZnO nanostructures have band gap of 3.37eV and excitation binding energy of 60Mev [2-4]. Also ZnO have peculiar properties such as near UV emission, transparent conductivity, piezoelectricity and electronic property. Due to these properties mentioned above ZnO have great potential applications in sensors, optoelectronics and solar cells. In order to tune the size, shape and properties of ZnO, various synthesis methods have been adopted [5-8]. Recently, researchers are focusing on the growth of ZnO with different morphologies like rod, tube, wire needles, flower and stars. In the present investigation, among the various synthesis method microwave assisted route have been chosen for the synthesis of ZnO nanostructures.

Because microwave assisted method have lot of advantages like 1) Reaction properties 2) Simple handling 3) Allowing simple and fast optimization of experimental parameters [9-15]. This paper reports the synthesis, structural and optical properties of microwave assisted ZnO nanostructures.

2. Synthesis Procedure

The synthesis strategy for ZnO nanostructure fabrication starts with the sequence of reactions. The precursors used for this investigation are Zinc acetate and liquid ammonia. Initially Zinc acetate was dissolved

in double distilled water to obtain 0.1molar concentration. The solution was stirred vigorously and the pH value of the solution was adjusted to 8 by adding liquid ammonia. This solution was placed in microwave oven and irradiated for 5, 10 and 15 minutes. The obtained products were filtered and washed with double distilled water. Finally the sample was dried and kept in furnace at 120°C for 5 hours.

The structural properties of ZnO nanoparticles were investigated using X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM) and the optical properties were studied using UV visible spectroscopy.

The average crystalline size (D) of the Zno nanostructures were calculated by using Scherrer's formula

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

Where
λ is the X-ray wavelength,
β is the full width at half maximum intensity of the diffraction peak located at 2θ
θ is the Bragg's angle.

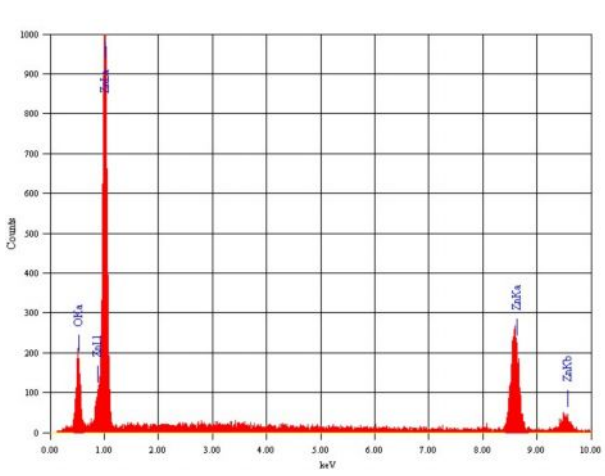
3. Results and discussion

3.1. EDAX analysis of ZnO nanostructures

The chemical composition of the ZnO nanostructure was obtained from the EDAX analysis. The chemical compositions of the synthesized ZnO nanostructure with different irradiation time are tabulated as shown in table (1). EDAX result showed that Zn content increases from 90.16 wt% to 92.89 wt% with increase in the microwave irradiation time. The maximum Zinc content of 92.89 wt% was obtained for ZnO nanostructures at the irradiation time of 15 minutes. The wt % of oxygen O decreases while increasing the microwave irradiation time.

Table 1: Results of EDAX analysis

S.No	Microwave irradiation time	Zn wt%	O wt%
1.	5 minutes	90.16	9.81
2.	10 minutes	92.19	7.81
3.	15 minutes	92.89	7.11



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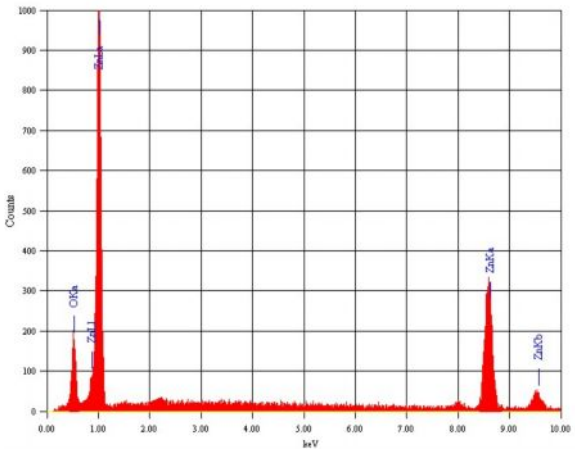

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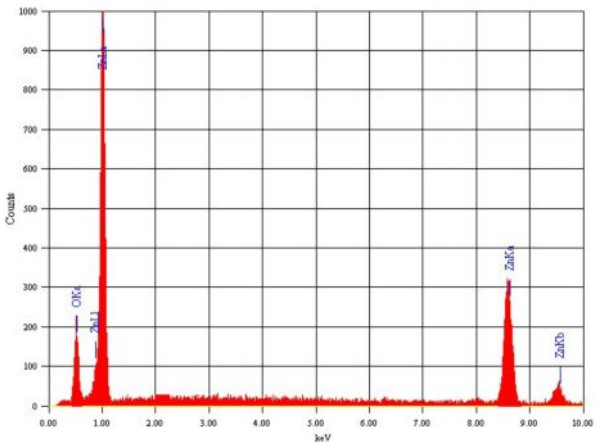
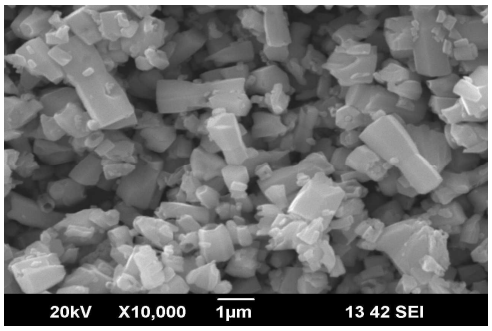


(c)

Figure 1: EDAX spectrum of ZnO nanostructures at irradiation time of (a) 5 min (b) 10 min (c) 15 min

3.2. Surface morphology of ZnO nanostructures

The SEM micrographs of ZnO nanostructures are shown in figure(2).SEM pictures of ZnO synthesized by microwave route clearly shows the formation of one dimensional nanostructures which may be regarded as nano rods. Among the three SEM pictures, figure c (microwave irradiation time of 15 minutes) reveals that the perfect formation of nanorods when compared to figure b (irradiation time of 10 minutes) and figure a (irradiation time of 5 minutes).This may be due to the irradiation power of microwaves on ZnO. As the irradiation time increases from 5 minutes to 15 minutes, ZnO nuclei starts to aggregate and leads to the formation of nano rods.The length of the nano rods ranges from 3 to 4 μm and the radius was about 30 - 35nm.



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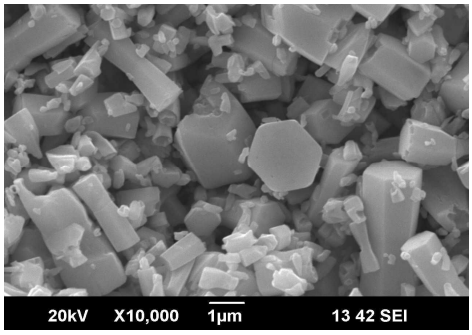

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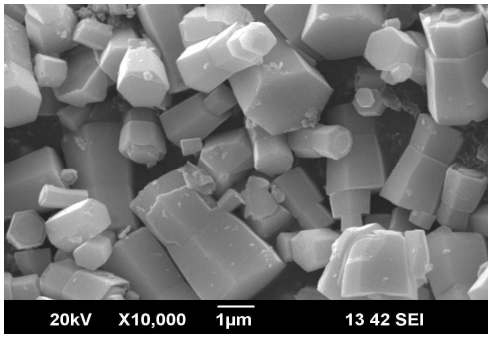


(c)

Figure 2: SEM pictures of ZnO nanostructures at irradiation time of (a) 5 min (b) 10 min (c) 15 min

TEM micrograph shows rod shaped ZnO nanostructures. The SAED pattern collected on the single nano rod is shown in fig (3).The bright spots can be attributed to single crystal of ZnO.

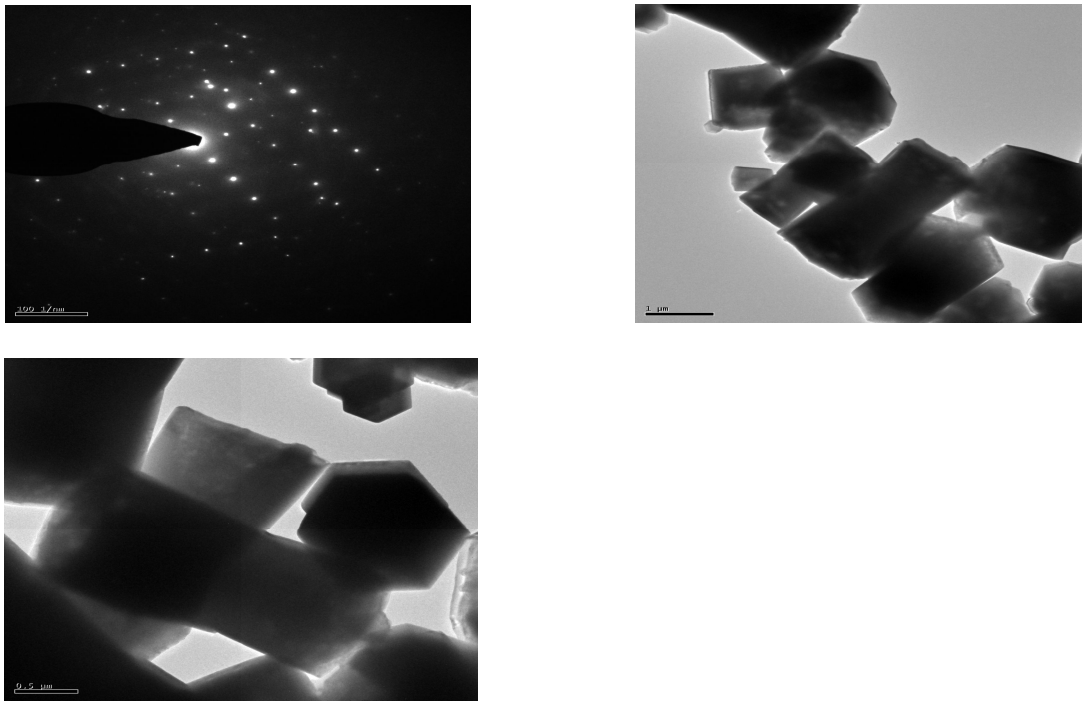


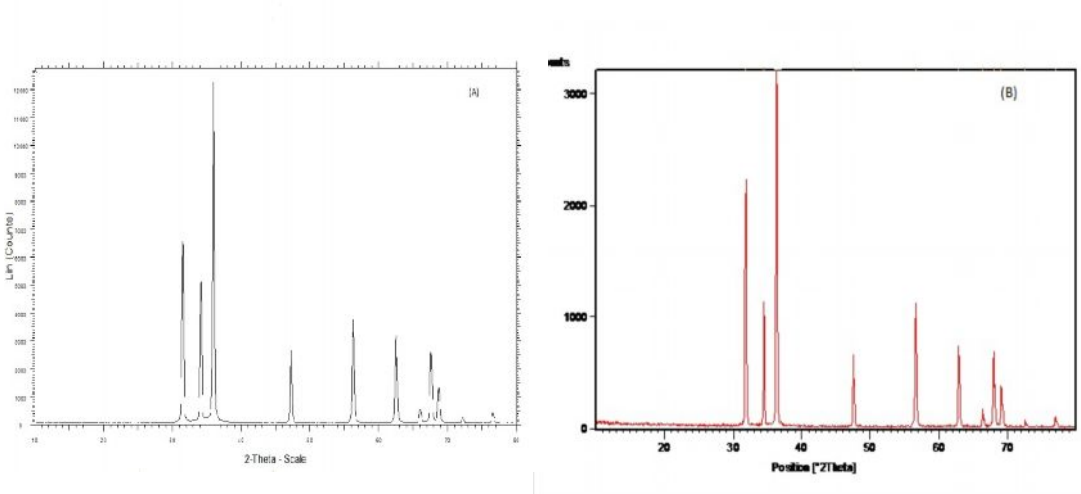
Figure 3: TEM pictures of ZnO nanostructures

3.3. Structural properties of ZnO nanostructures

The structural properties of ZnO nanostructures were analyzed by using X-ray diffraction patterns and are tabulated as shown in Table 2. The obtained XRD patterns of ZnO are shown in figure (4).

Table 2: Results of XRD analysis

S.No	Irradiation Time	2θ	d-spacing θ	Particle Size	Stress
1.	5 minutes	35.922	17.961	28.51nm	81.2
2.	10 minutes	36.190	18.095	31.16 nm	76.09
3.	15 minutes	36.278	18.139	35.44nm	66.03



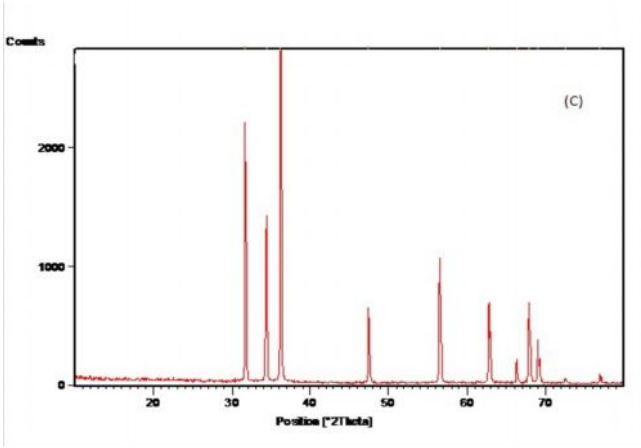
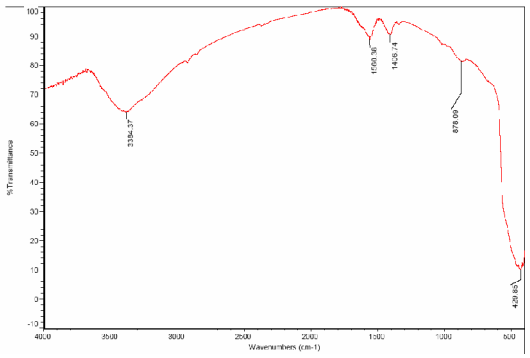


Figure 4: XRD patterns of ZnO nanostructures at irradiation time of (a) 5 min (b) 10 min (c) 15 min

From Table (2), as the irradiation time increases, the particle size increases which shows only for minimum microwave radiation, the particle size reduces. Stress is the parameter which reduces when irradiation time increases.

3.4 FTIR analysis of ZnO nanostructures

The FTIR spectra of the samples were recorded over 400 – 4000cm^{-1}. In all the cases the FTIR spectrum shows absorption bands centered at about 3409 and 3417 cm^{-1}. This can be assigned to the O-H stretching vibrations and bands centered at 1555 and 1560 cm^{-1} that correspond to the asymmetric and symmetric C = O stretching modes (CO_2 modes).



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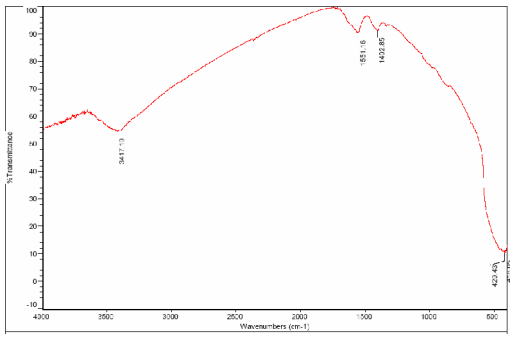

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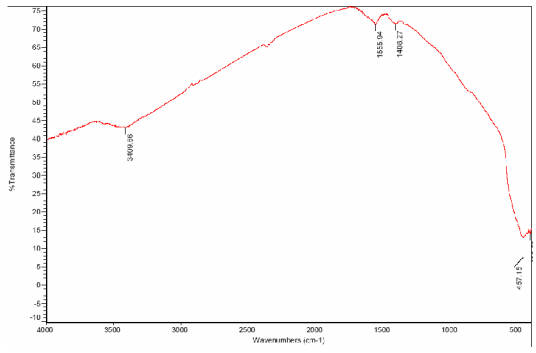

(c)

Figure 5: FTIR Spectra of ZnO nanostructures at irradiation time of (a) 5 min (b) 10 min (c) 15 min

3.5. UV analysis of ZnO nanostructures

The optical constants of microwave irradiated Zinc oxide nanostructure samples a, b& c were determined by UV-Visible spectra.

From the fig (6), as the irradiation time increases the band gap value decreases and the absorption edge ranges from0.83 to 1.20.

Table 3: Results of UV analysis

S.No	Irradiation Time	Absorption Edge	Band Gap (eV)
1	5 minutes	0.83	3.50
2	10 minutes	1.20	3.30
3	15 minutes	0.93	3.10

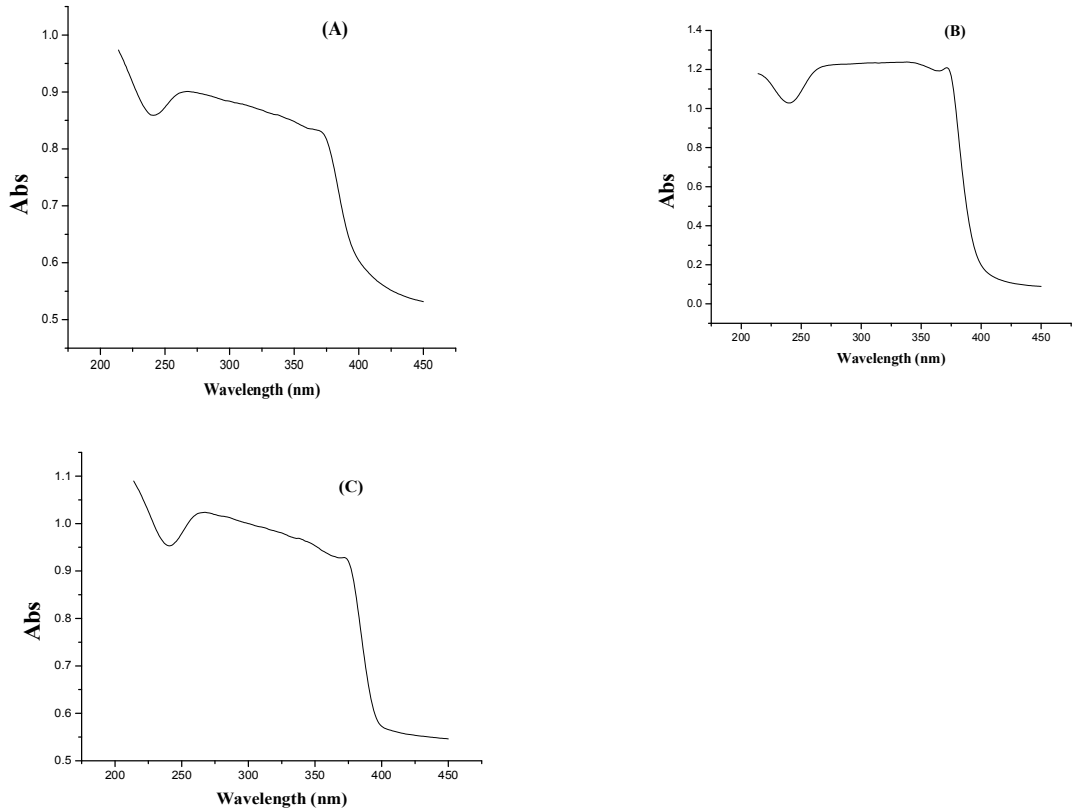

Figure 6: UV Spectra of ZnO nanostructures at irradiation time of (a) 5 min (b) 10 min (c) 15 min

4. Conclusion

The ZnO nanostructures were successfully synthesized by microwave assisted wet chemical technique at different irradiation time (5,10 and 15 minutes).The morphological studies of ZnO nanostructures reveals that an increase in irradiation time generates more defined structure with particle size in the range of several nanometers. It was observed that the formation of nanorods is favored as the microwave irradiation time increases.HCP was the dominant structure of microwave assisted ZnO nanostructures with average particle size of 28.51nm.The band gap value of ZnO nanostructures is found to have 3.10eV at irradiation time of 15 minutes. In summary, results obtained indicate that the microwave assisted method is a promising low temperature, cheap and fast method for the production of ZnO nanostructures.

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