National Conference on Recent Trends in Synthesis and Characterization of Futuristic Material in Science for the Development of Society
(NCRDAMDS-2018)
In association with
International Journal of Scientific Research in Science and Technology

Synthesis and characterization of SnO_2-ZnO composites nano particles.

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ABSTRACT

The nanocrystalline SnO_2-ZnO composites is prepared by microwave assisted technique.A stannous chloride is used as a precursor with deionized water as a solvent. The morpholozy and nano structure have been characterized by X-ray diffraction (XRD),scanning electron microscopy (SEM),energy dispersive analysis by x-ray (EDAX),transmission electron microscopy (TEM) and selected area electron diffraction (SAED).it has been shown that ,spherical nano crystalline sno_2-zno nano composite is formed.

I. INTRODUCTION

As an important semiconductor, tin dioxide (SnO_2) has been intensively explored with regard to its synthesis and application . SnO_2 is stable n-type wide band gap semiconductor (Eg – 3.7 Ev) is well known for its potential application such as gas sensor ,dye based solar cell, photochemical and photoconductive device, liquid crystal display[1-3], opto electronic devices [4-5] electrode material [6] and catalyst [7]. Zinc oxide (ZnO) is also the important wide band gap semiconductor (Eg – 3.37 Ev.) and large binding energy at room temperature, which has attracted much attention due to its unique properties. ZnO has found numerous application such as gas sensor (8),transperant electrode (9),PH sensor (10),biosensor(11),acoustic photo devices (12) and UV – photodiodes (13).

One of the effective method for obtaining desired material with modified properties is mixing of two different semiconductors with appropriate conduction and valance band edges (14).With coupling two metal oxide nano particles, better structure with more advantages for sensor application can be achieved. It has been demonstrated that the combination of SnO_2 and ZnO hetero junction nanostructure, such as core shell, tetra pods and wire-dots can greatly enhance performances that their individual can never have (15). The nanocrystalline SnO_2- ZnO composite have been synthesized by an in-situ electro spinning technique (16-17), double layer thin films by sol-jel. two stage chemical deposition method(18).core-shell nanowires by two-step vapour phase deposition method (19) and nano composite by hydrothermal method (20).In comparison with all above mentioned routes microwave technique is more advantageous for following reason, rapid volumetric heating, cost effective, technique is more compact, require smaller space, microwave energy is precisely controllable ,turn on or off instantly. This increases the production run time, microwave energy is selectively absorb thus yield is improved.

This investigation, report the synthesis and characterization of SnO_2-ZnO nano composite sample using microwave radiation with 2.45 GHz frequency. Crystalinity, morphology and structure of microsphere was investigated, followed by discussion of possible formation mechanism. SnO_2-ZnO nano composite is formed after heat treatment given to SnO-ZnO composite.

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II. EXPERIMENTAL

AR grade stanous chloride from merk ltd, AR grade zinc chloride (merk ltd.) were used as a precursor. AR grade diluted ammonia solution is used as a base. The Stannous chloride was separately diluted with de-ionized water with simultaneous stirring. The PH of the solution was maintained by using liquid ammonia diluted with water. Similarly Zinc chloride solution was separately diluted with de ionized water with simultaneous stirring. The PH of the solution was also maintained by using liquid ammonia diluted with water. The stannous chloride and zinc chloride solution was mixed with each other and stir with magnetic stirrer for half an hour. The resultant precipitate then wash with de ionized water more than ten times, until chlorine ions are removed. The chlorine free precipitate was then irradiated with microwave energy using Samsung house hold microwave oven for optimum time. The radiation frequency was 2.45 Ghz. and its power up to 1 kwt. The resultant mixture of SnO – ZnO composite nano particles were then sintered at 400 ^{0} C. For 5 an hour and their structure was analyzed by x-ray diffraction pattern (XRD) using Cukα wavelength of 1.54 AU. The morphology of the sample was studied by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).The elements and their percentage in the resultant nano crystalline sample was observed by energy dispersive analysis by x-ray (EDAX).The crystallinity of the composite sample was studied by selected area electron diffraction (SAED).

III. RESULT AND DISCUSSION

The XRD pattern of SnO_2 – ZnO nano composite sample sintered at 400 ^{0} C is shown in fig.1 .The ppt. before microwave treatment will contain hydroxyl group, after microwave treatment SnO-ZnO composite will form .This shows that microwave radiation transform hydroxyl group into oxide group. The formation of SnO_2 from SnO will start at sintering temperature 300 ^{0} C (21) for more than four hour but the ZnO remains as ZnO because large amount of thermal energy is required to transform ZnO into ZnO_2 ie. the thermal energy produced at 400 ^{0} C sintering temperature is not sufficient to oxidized ZnO into ZnO_2.Therefore ZnO remains as ZnO even after sintering the microwave radiated sample at 400 ^{0}C for five hour, but SnO transform into SnO_2.thus the formation of SnO_2-ZnO nano composite sample takes place after sintering the microwave radiated SnO-ZnO crystalline particle at 400 ^{0}C for five an hour.

XRD pattern exhibited very sharp diffraction peak because of high crystallinity of the sample .further the broadening of full width at half maxima of the intensity peak in the XRD pattern shown the formation of crystalline particle in the nano range. in the XRD pattern both the peak of SnO_2 and ZnO are observed in the XRD pattern which confirmed the formation of SnO_2-ZnO composite nano particles. It is also manifested by EDAX study of the sample .The typical diffraction peak at 2θ of 26.591 ^{0},33.888 ^{0},37.959 ^{0},38.991 ^{0},42.645 ^{0},51.787 ^{0},54.767 ^{0},57.855 ^{0},61.892 ^{0},62.618 ^{0},64,768 ^{0} and 65.981 ^{0} were indexed with corresponding millar indices (110) (101), (200), (111), (210), (211), (220), (002), (310), (221), (112) and (301) which could attributed to cassiterite tetragonal SnO_2 structure. all typical diffraction peaks measured in 2θ range corresponds to tetragonal structure of SnO_2 with lattice constant 4.736 and 3.185. These peakes are in good agreement with those on standard JCPDS card number 072- 1147. The peak corresponding to smallest (2θ) attributed to $ZnSnO_4$ (22)

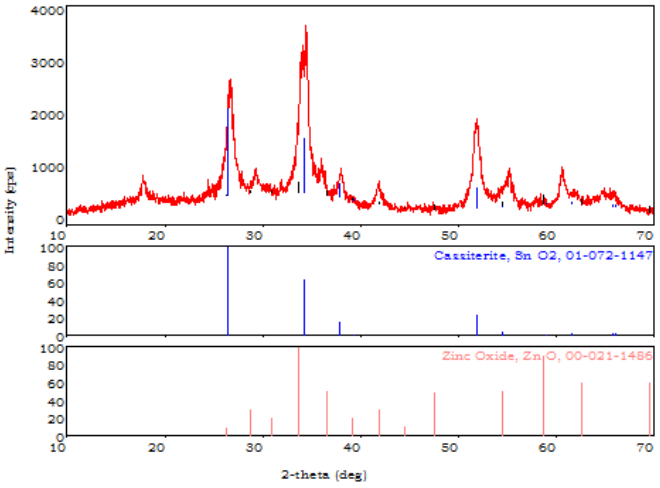

Figure 1. XRD pattern of SnO_2-ZnO nano composite

The XRD peaks of ZnO are as 26.26 ^{0},28.68 ^{0}, 30.91 ^{0}, 33.66 ^{0},36.49 ^{0},39.135 ^{0},41.989 ^{0},44.6 ^{0},47.569 ^{0},54.582 ^{0},58.765 ^{0},62.728 ^{0} and 69.583 ^{0} which match with jcpds cards number 021 – 1486.thus the diffraction pattern confirmed as SnO_2-ZnO nano composite sample. The average crystalline size of the nano particle was evaluated using scherrer formula.

$$d = k\lambda/\beta\cos\theta$$

where- d is the crystalline size, k is grain shape dependent constant (0.9),λ is the wavelength of incident x-ray beam (1.54 A^0).θ is the brags reflection angle and β is full width at half maxima. The average particle size from peak of SnO_2 in XRD pattern of SnO2-ZnO sample is found to be in the range 11 nm. and that from ZnO peak it was 10 nm.

Figure 2 shows the SEM micrograph of SnO_2-ZnO coposite nanoparticles prepared by microwave assisted technique sintered at 400 ^{0} C. As shown in fig. 3,the as-synthesized sample consist of mixture of fine tiny spherical and also leafy and platy nano particles were observed. The nano particles revealed to be well crystalline and slightly agglomerated spherical nano particle in the size range of 11.4 nm., which agree with those estimated from XRD pattern using Scherrer formula.fig .3 shows the EDAX pattern of SnO_2-ZnO nano composite particle sintered at 400 ^{0} C. The sign of O, Sn and Zn are observed in the pattern therefore the sample was compose of 40.57 % - Zn,39.64 % - O and 19.79 % -Sn which clearly support that the synthesized sample is SnO_2-ZnO composite nano particle.

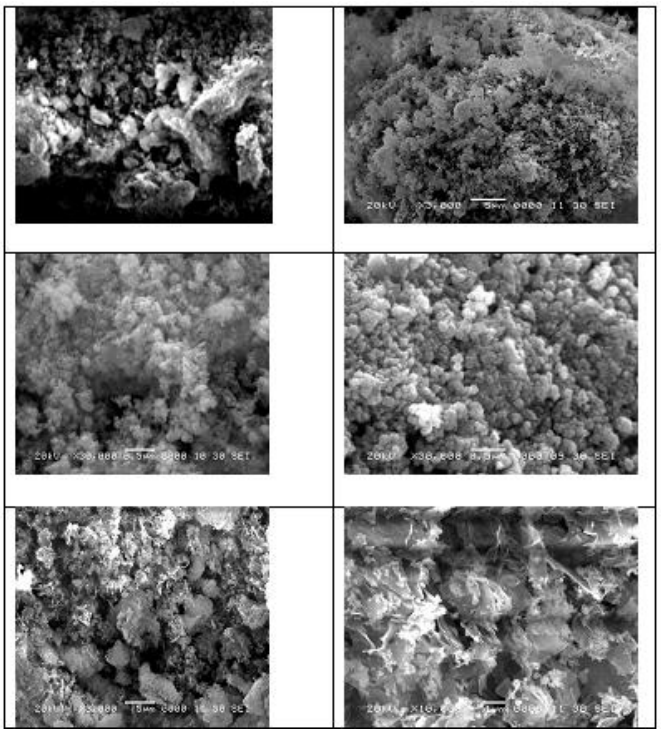

Figure 2. SEM micrograph of SnO_2-ZnO nano composite

Figure 4 shows the TEM micrograph of the SnO_2-ZnO nano composite sample. The particle size of SnO2 -ZnO composite sample shown in the TEM micrograph is in good agreement with the result of SEM micrograph. particle size observed in TEM micrograph match with those calculated from XRD pattern. Rod like particle are attributed to ZnO while spherical quantum dot are of SnO_2. SAED pattern is shown in fig. 5. The selected area electron diffraction pattern of the sample indicates the crystalline nature of the sample.

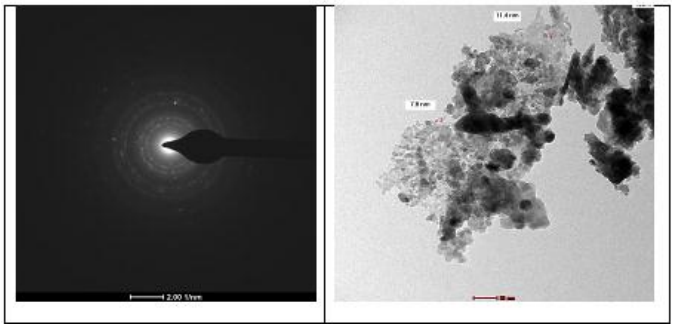

Figure 3.TEM micrograph of SnO_2-ZnO nano composite

IV. CONCLUSION

The nano crystalline SnO_2- ZnO composite sample has been successfully prepared by microwave assisted co-precipitation method sintered at 400 ^{0}C using di hydrated stanous chloride and zinc chloride as precursor and de ionized water as a solvent. This process is inexpensive, time saving, compact and more convenient method to synthesize the nano particle for the application in nano sensor, optoelectronic devices, electrode material, PH sensor, biosensor and other industrial application.

V. REFERENCES

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