International Journal of Advance Research in Science and Engineering
Volume No.06, Issue No. 11, November 2017
www.ijarse.com
IJARSE
ISSN: 2319-8354

Synthesis and Characterization of ZnO Nanostructures By Oxidation Technique

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ABSTRACTS

We report simple methods to synthesize Zinc oxide nanostructures, without using catalysis with less complicity. This was done by oxidation of zinc foil at various temperatures (200^{o}C-1000^{o}C) and various times (1-3h) and also we find that the nanostructures size and shape depends on heating rate, temperature and heating time. Zinc oxide plays on important role in current industry due to its special characteristics such as anti corrosion, anti bacteria, has low electrons conductivity and excellent heat resistance. Therefore, the objective of this study is to synthesize zinc oxide nanostructures with the most practical ways by using oxidation technique.These oxide structures were characterized by SEM, EDX, and DSC.

Keywords: DSC,EDX,Oxidation,SEM,ZnO,

I. INTRODUCTION

ZnO is one of the few dominant nanomaterials for nanotechnology and zinc oxide belongs probably to the biggest group of one dimensional nanostructures. Zinc oxide (ZnO) has a wide direct band gap (3.37 eV) and a relative large excitation binding energy (60 meV) compared to thermal energy (26meV) . A wide band gap has many benefits like enabling high temperature and power operations, reducing electronic noise, making sustenance in large electric fields possible and raising breakdown voltages. By proper alloying with MgO or CdO, the band gap can be tuned in the range of 3-4 eV. ZnO is also called as II–VI semiconductor, because Zn belongs to II group, and O_2 belongs to VI group in the periodic table. ZnO exhibits the most splendid and abundant configurations of nanostructures that one material can form. Owing to its unique properties and potential application in solar cell, electro and photo-luminescence devices, chemical sensors and so on, ZnO becomes an attractive inorganic material. ZnO with hierarchical structure has fundamental importance to understand the growth habit of ZnO crystal; moreover, considering their high surface to volume ratio, they are of great physical or chemical activities in gas-sensor and photo-catalysis. Zinc oxide (ZnO) received much attention because of its unique piezoelectric properties made suitable for surface acoustic wave devices, optical fibers and up to electronic devices. Due to the high optical band gap ZnO films have been used as window layers in copper indium diselenide based hetero junction solar cells to enhance the short circuit current.

II. EXPERIMENTAL

There are many methods to synthesize ZnO nanostructures such as chemical vapor deposition physical vapor deposition and molecular beam epitaxy, ZnO nanowires have been synthesized simply by heating Zn powders containing catalyst nanoparticles , optically pumped nanowires . The vapor–liquid–solid (VLS) mechanism is responsible for the nano-wire growth, in which a metal or an oxide catalyst is necessary to dissolve feeding source atoms in a molten state initiating the growth of nano-materials. However, most of the methods create ZnO nanostructures by using a catalyst, but there are some methods to form ZnO nanostructures without using catalyst, one of such method is ZnO nanorods formation by Zn oxidation is simple method and without complexity. The oxidation method is most attractive method because from this method we can synthesize different type of nanostructure

Synthesis of ZnO Nanostructure by Oxidation Technique:

In this experiment pure Zn (foil or powder) was taken in silicon crucible, the crucible size is average 1.5cm in diameter (cone shape) 2.5cm in depth. The crucible which contain a Zn (foil or powder) is put in Furnace, and switch on the furnace the temperature reach to say ten temperature (200^{o}C, 300^{o}C, 400^{o}C, 500^{o}C, 600^{o}C, 700^{o}C, 800^{o}C and 900^{o}C) and hold the temperature for 1 and 2 hour then switch off the furnace cool the Zn (foil or powder) by annealing after cooling the Zn color is change in to gray color it initial color is white color that indicate the ZnO nanorods is formed. This nanorods was characterizes by JEOL JSM-6480LV scanning electron microscope, EDX, DSC and XRD.In this experiment we oxides both Zinc foils and Zinc powder at wares temperatures (200^{o}C, 300^{o}C, 400^{o}C, 500^{o}C, 600^{o}C, 700^{o}C, 800^{o}C and 900^{o}C) and wares times (1 or 2 hours). We oxides the zinc in the presents of common air atmosphere, for this we use muffler furnace.

III. RESULTS

3.1.Synthesize of ZnO Nanostructures by Oxidation Techniques:

Pure Zn foils were oxidized at temperatures 200^{o}C, 300^{o}C, 400^{o}C, 500^{o}C, 600^{o}C, 700^{o}C, 800^{o}C and 900^{o}C and for various periods of time. We take Zn foils in silicon crucible. This silicon crucible is kept in a muffle furnace and heated at different temperatures and different times. The oxidized samples are analyzed using SEM, EDX and XRD. First we observed that Zn has a metallic lusture but after heating, the lusture of metallic Zn is lost and it starts to have a white coloured layer on it which is the typical colour of ZnO. This color change indicates the formation of ZnO on the metallic Zn surface.From SEM images we analyze that the formation of ZnO nanostructures depends on heating temperature and also the holding time. If heating temperature is high it indicates oxidation is more so nanorods is more likely to form but in the lower temperature range of 500-600^{o}C the ZnO structure formed is mainly nanorods, nanobelts, nanorings, nanoribbons nanoneedles, nanolaves and nanowires etc. Heating time also affects the ZnO structure. We also find ZnO nanostructure forming temperature at around 400^{o}C (near melting temperature 419^{o}C) below this temperature at 200^{o}C and 300^{o}C we do not find any oxidation of Zn foils taking place. The DSC results in Fig. 4.3(c) also reflect the same. Oxidation of Zn foil mainly starts from 200^{o}C or beyond.From SEM image we not find ZnO nanostructure at 300^{o}C. It indicates that the Zn is not oxidized at 300^{o}C. The EDX analysis indicates that the Zn foil is 100 % Zn

International Journal of Advance Research in Science and Engineering
Volume No.06, Issue No. 11, November 2017
www.ijarse.com
IJARSE
ISSN: 2319-8354

even after oxidation of the Zn foil at 300°C for 2 hours. Nanostructures are forming on the Zn foil at temperatures beyond 400°C

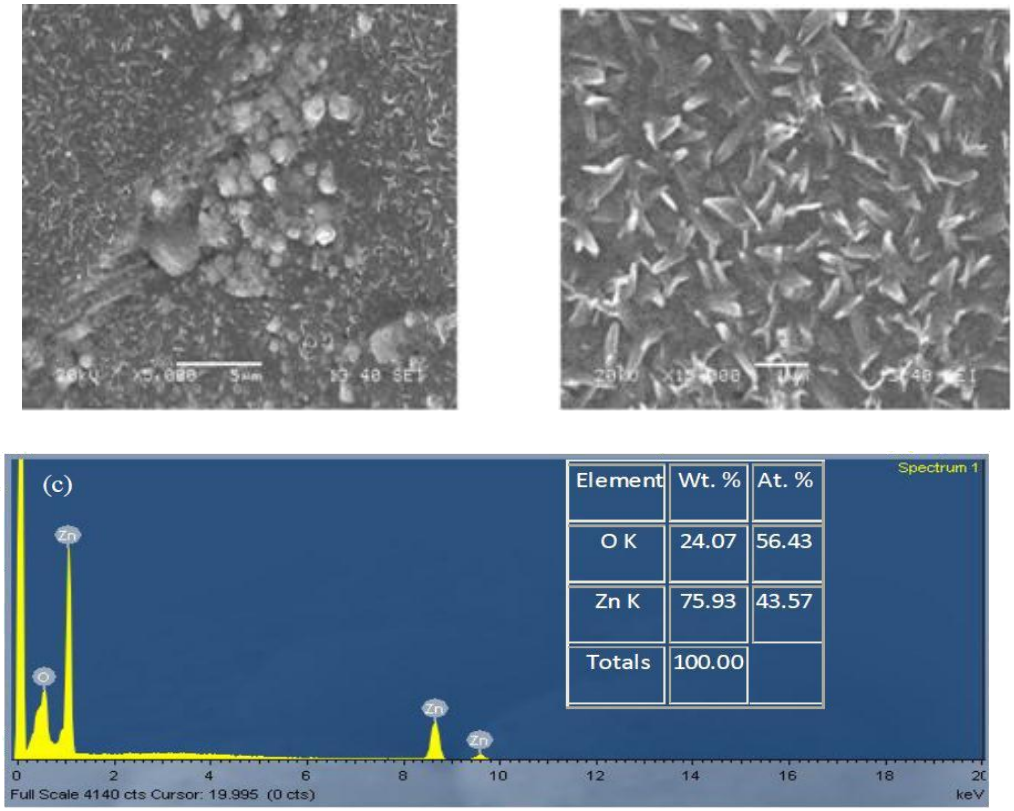

Fig:1).SEM image of Zn foil after annealing at 400oC for 2 hours. In this we are find ZnO nanorods. It means nanostructures are start to form from 400° C

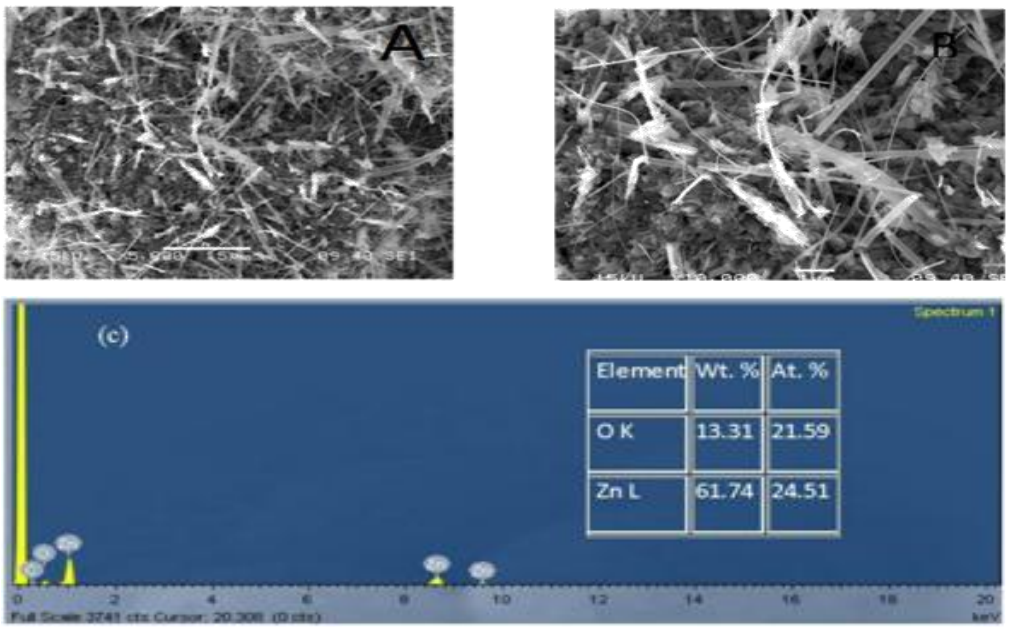

Fig.2).SEM images showing ZnO nanostructures obtained on Zn substrate after holding at 500°C for 2 h. (c) EDX of the obtained sample

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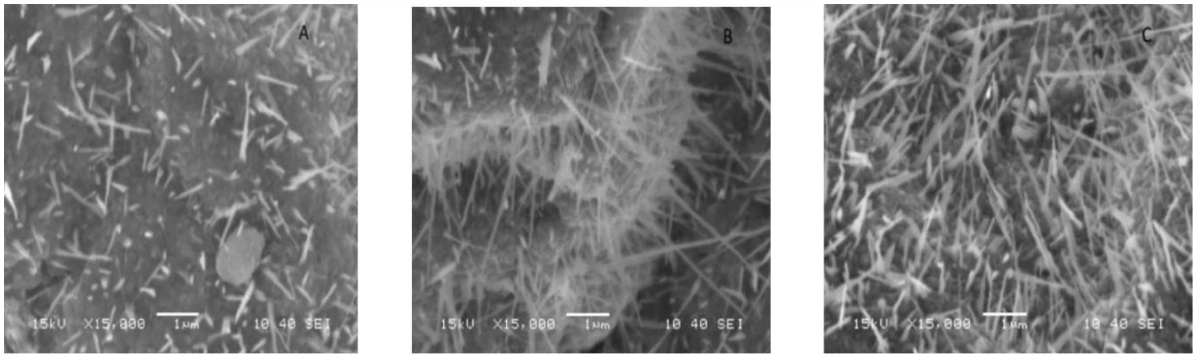

Fig.3).SEM images of Zn foil after annealing at same temperature (600^{o}C) at different times (a) for 1 h, (b) for 2 h and (c) for 3 h.

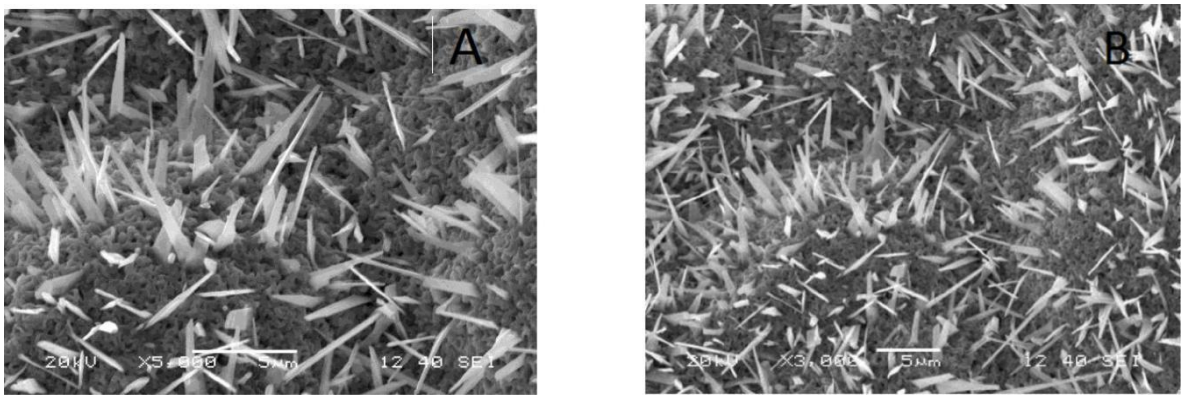

Fig.4).SEM images of ZnO microstructures obtained on Zn substrate holding Zn at 700 °C for 2 h. (c) EDX of the protruded structure.

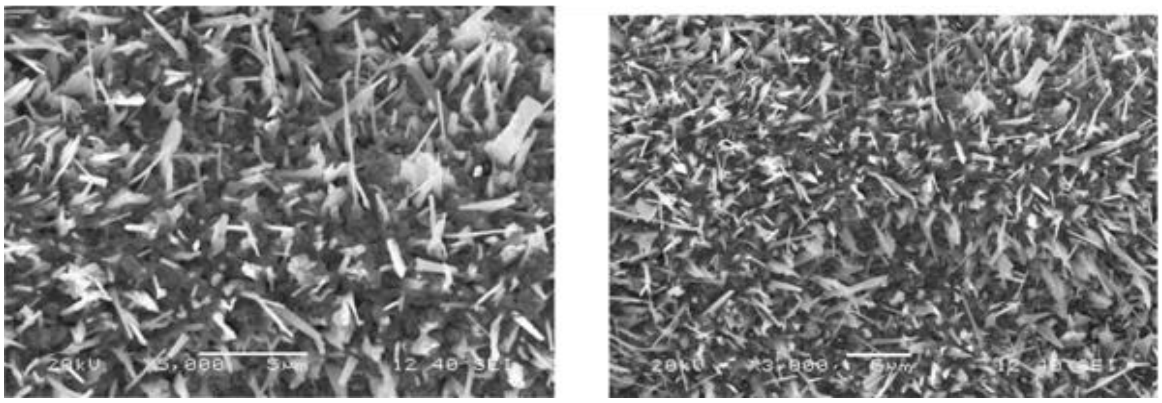

Fig.5).SEM images showing ZnO structures obtained on holding Zn for 2 h at 800°C.

From above figure 2(c) and 4(c) we absorbed that oxygen percentage is increasing with heating temperature. Here we find that nanorods and nanoneedles like nanostructures this nanorods and nanoneedles are increasing with temperature. Temperature is high nanorods height is more and according to height width is decreasing from bottom to end tip. And also find that the number of nanorods is increasing with temperature. The nano rods and nanoneedles are formed in triangle shape and different nanorods in different height.From above figures we analysis that ZnO nanostructures are depending on heating time. Heating time is more ZnO nanorods are more and height is increase with time width of nanorods are decrease. Heating time increase the shape of nanorods and nanoneedles come into triangular shape rods. But nanostructure mainly depends on heating

temperature.From the SEM image in Figs. 1(a-b) we can confirm that at the oxidizing temperature of 400^{o}C the surface of Zn shows the formation of ZnO structures. When Zn was held at 500^{o}C for 2 h the SEM images of the surface shows the formation of a wide range of ZnO nanostructures Figs. 2(a-b)). EDX analysis of these nanostructures in Fig. 2(c) reveals that their composition is almost that of stoichiometric ZnO (21.59 at. % O and 24.51 at. % Zn). At the oxidizing temperature of 600^{o}C also we see that the surface of Zn is completely filled with nanostructures of ZnO (Figs.3 (a-b)). As can be seen from the SEM image in Figs.3(a-b) there is a wide range of nanostructures of ZnO on the Zn surface. ZnO having structures such as nanowires, nanobelts and nanoribbons could be seen in the samples oxidized at 500 and 600^{o}C (Figs. 2(a-b) and Figs. 3(a-b)). On the other hand the oxidation of Zn at 700^{o}C does not show nanostructured ZnO (Figs. 4(a-b)). There is a gradual increase in the size of the ZnO structure with further increase in temperature. Figs. 5(a-b) are the SEM images of Zn oxidized at 800^{o}C for 2 h. Nanostructures of ZnO could not be seen at this oxidizing temperature. The EDX analysis in Fig. 4(c) of the ZnO microstructure formed by oxidizing Zn at 700^{o}C for 2 h shows that the at. % of Zn is 43.57 % and that of O is 56.43 %, suggesting that highly stoichiometric ZnO is formed at this temperature. The EDX spectrum shows that only O and Zn elements are detected, confirming the formation of pure ZnO

3.2.DSC/TG Analysis Of The Pure Zinc Sample:

The DSC/TG analysis of the pure Zn sample was done in order to find out the temperature at which oxidation of Zn starts and weight gain during oxidation as a function of temperature. The TG curve in below Fig.6.) Shows a gain in weight by 1.09 %. At temperatures above 200^{o}C there is sign of weight gain which is due to the oxidation of Zn. At temperatures above 200^{o}C and up to 450^{o}C a weight gain mainly due to the oxidation of Zn could be seen. Below 200^{o}C hardly any gain in weight of Zn could be found. At 416.8^{o}C an endothermic peak corresponding to the melting temperature of Zn can be observed in the DSC plot.

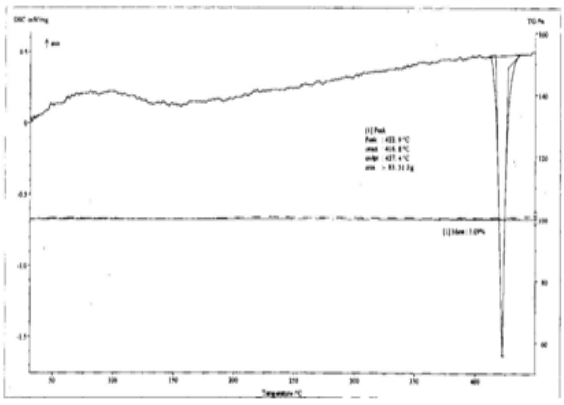

Fig.6). Graph shows DSC/TG analysis of pure Zn foil in range up to 450^{o}C (30^{o}C/10 min) in air.

IV.CONCULSIONS

A Simple technique for ZnO nanostructure synthesis has been used here successfully. Different ZnO nanostructures are synthesized by simple techniques for different applications. Here we use oxidation technique, it is very simple, no complexity and without catalyst technique to synthesis a wide range of ZnO nanostructures.

We also find that the nanostructure density depends on heating time and temperature. Oxidation technique is very simple and low cost method to synthesize the ZnO nanostructures

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