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Making a nanosensor sensitive to CO gas with SNO_2 Nanopowder prepared by One-Step Solid State synthesis method

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ABSTRACT

In this work,SnO2 nano powder is prepared by One-Step solid state synthesis method and beside physical and crystalline characterization of the nano powder in various temperatures ,it was used for making a sensor sensitive to Carbone monoxide gas.

The stages of preparation and making the needed powder, manufacturing of the sensor including inner layer , heater, placing the layer, Electrodes are investigated in this paper. Then the sensitivity and stability of the sensor is studied using various diagrams.

Nano particles of SnO2 are prepared using the primary powders of CaCl2,Ca(OH)2,SnCl4 .5H2O with the weight ratio of 2:1 for Sn/Ca in sizes of 3 to 68 nanometers and are heated from 200 to 1000 Celsius degrees. using FTIR,SEM, XRD and TGA ,the morphology and nano structure of SNO2 particles were checked and used for making the nano sensor finally.the manufactured sensor was examined by different concentrations of Carbone monoxide and diagrams of sensitivity and stability was studied in various temperatures. In addition to the advantage of simplicity and low cost of the process,the nano particles of SnO2 made in 400C with the size of 7 nanometers are used in nno sensor and acceptable results for all three requirements of a sensor(sensitivity, selectivity and stability) was achieved .

KEYWORDS:SnO2 nanoparticles, One-Step solid state, nano sensor

1. INTRODUCTION

In the last two decades, the technology of materials and machines with very small size is achieved and the will lead to a extraordinary revolution which will change the world of science and technology.

Technology and engineering in coming century will work with instruments and products which have ultra small dimensions. presently, processes with the dimensions of a few molecules is designable and controllable.

Also, the mechanical, chemical, electrical magnetic and optical properties of materials in layers in the dimensions of nanometers can be understood, analyzed and measured.

One of the pioneer fields in nano materials is research and development of production methods, synthesis of nano particles and introducing them to industrial usage. in nano particles and in nano structure materials as a whole, the small size of particles creates some peculiar mechanical, chemical, electrical magnetic and optical characters.

Nano particles of SnO2 are a kind of ceramic nano particles and have various applications in optic and opto electronic apparatus and instruments and gas sensors because of their unique characteristics such as good conductivity, optical transparency in visible region, high porosity, good heat stability and mechanical strength .[1]

Seiyama el al retrieved that the conductive urface of a semi conductive materials like SnO2, TiO2 and ZnO canbe effected by ambient gases so the conductiver sensors of semi conductors were made based of theis fact.In general, gas sensors are consist of a sensitive layer and a signal transfer transformer .The feedback of this sensors are based to this fact that with achange in the composition of ambient atmosphere ,one of the specification of the layer like absorbsion, mass,capacity or it's resistance changes and this change is transformed to a signal and can be senced and measured.

Concerning to the increasing environmental pollution caused by petroleum product combustion in cars and factories ,the need of humans for measuring and controlling the pollutin seems a urgent and important necessity.from this, the gas sensors are a good substitute for expensive and complicated sensation systems due to their simplicity ,high sensitivity ,small size and low cost. [2]

Based on above mentioned items, SnO2 nano powder is prepared by One-Step solid state synthesis method and beside physical and crystalline characterization of the nano powder in various temperatures ,it was used for making a sensor sensitive to Carbone monoxide gas.

MATERIALS AND METHODS

Preperation of SnO2 nano particles using CaCl2 and Ca(OH)2 powder

After making the initial powders according to table 1 , SnCl4.5H2O is mixed with CaCl2 and are grinded for 30 minutes to make sure that the combination is done. Then Ca(OH)2 is added to the powder in the mill and

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is grinded for another 30 minutes. The emerge of water on the upper walls of the grinding apparatus after adding Ca(OH)2 shows that the combination is happened .So by adding distillated water to the powder and primary centrifuging the product in 8000 RPM for 10 minutes in room temperature and decanting it according to reaction 1 ,we can make sure that the salt is taken out of the product.

Reaction 1 SnCl4.5H2O (s)+Ca(OH)2 $\longrightarrow$ CaCl2(s)+SnO2.H2O(S)+2H2O

Reaction 2 $Ag(NO_3) + CaCl_2 \longrightarrow AgCl + Ca(NO_3)_2$

Observing white precipitation implies the existence of salt in final product. In this case ,the centrifuging is repeated with the same conditions. From experience, this can be achieved by seven set of centrifuging action.

Table 1. Spcification of basic powders for making SnO_2 nano particles

Nano Powder	Weight(gr) - mole	Chemical formula	Sn/Mg ratio
SnO_2	7 (0.02 mole)	$SnCl_4$.5 H_2O	2:1
	1.11 (0.01 mole)	$CaCl_2$	
	2.96 (0.04 mole)	$Ca(OH)_2$	

For investigation on the effect of heat and examination of their heat stability ,they were annealed for 4 hours in 200,400 ,600,800 and 1000 Celsius degree and were cooled with the same rate.

Study of nanostructure of SnO2 Powders using XRD analysis

According to XRD images (Fig. 1.)by comparing the peaks in (101),(110) and (200) with the standard reference, the existence of rotil tetragonal SnO2 was proved in temperatures 200,400,600,800and 1000, it can be seen that the powder annealed in 400 ^{o}C has good particle size and less variations in size.In higher temperatures some peaks are increased in upper angles which corresponds with the reference samples.[3]

The size of nano particles(D) was calculated by the Schorrer formula (equation 1)and is reported in tables 2.

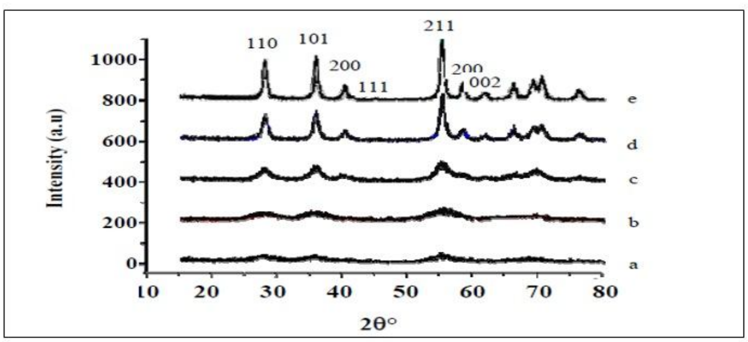
In the equation, the wave length of the used X ray is λ=1.541 A , β is the width of maximum peak and θ is the diffraction angle.

$$D=\frac{0.9\lambda}{\beta\cos\theta} \qquad \text{Equation 1}$$

Table 2. The effect of temperature on the particle size of prepared Powder

Annealig Temperature ^{O}©	200	400	600	800	1000
size of nanoparticle	3.36	6.69	41.23	68.00	32.00

Fig. 1. Comparison of XRD diagrams of SnO_2 in different temperatures



a)200 ^{o}C b)400 ^{o}C C)600 ^{o}C d)800 ^{o}C and e)1000 ^{o}C

TGA Thermal Analysis of SnO2 Nano powder

By TGA,the decrease trend in the weight of SNO2 nano particles ,prepared for annealing in 400 C was studied according to the Fig. 2. According to the curve 5 in Fig. 2 ,there is a endothermic peak in 80C which shows a loss in the weigth of the sample by 3 percent.

It sees that this loss in the weight is due to evaporation of the surface water and is continued to 450 C. after that, the weight is constant and from 450C , the SnO2 nano particles are stable.

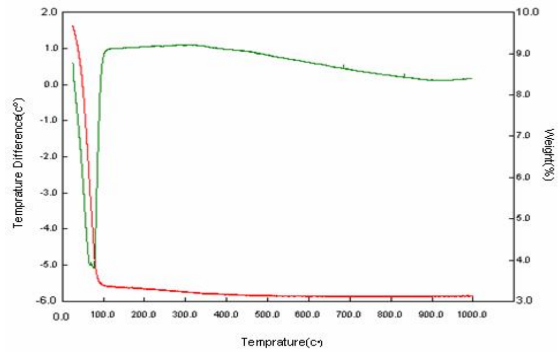


Fig. 2. TGA for SnO2 nanoparticles in 400 °C for 4 H

FTIR and SEM analysis of SnO2 Nano particles

According to XRD and TGA Analysis ,the nano particles prepared in 400 C for 4 hours have the maximum stability ,as well as the smallest size. Consequently, this powder was selected for making the sensor due to the positive effect of small size of the particles on high sensitivity of sensors.

SEM images of the samples annealed in 400 Celsius degree witht the power of 40,000 and 80,000 is shown in Fig. 3.

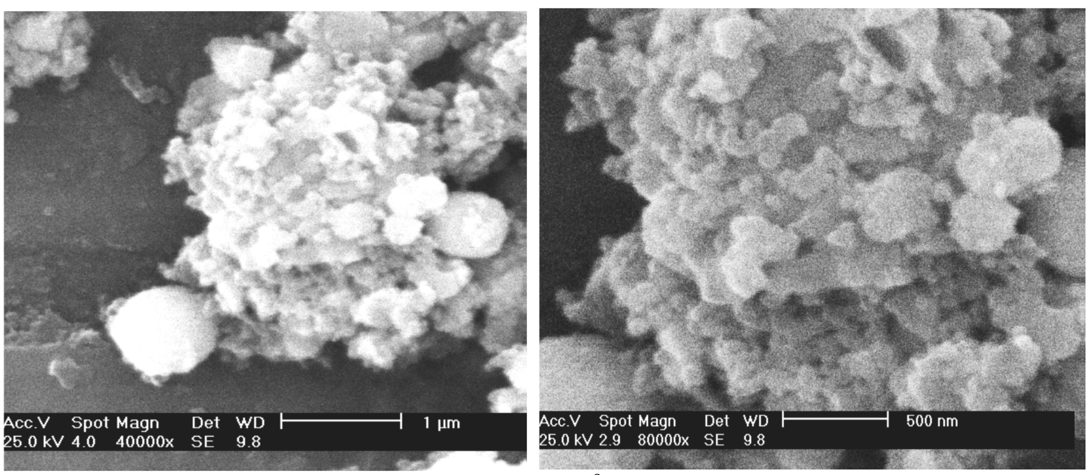


Fig.3. SEM image SnO2 nanoparticles in 400 °C witht the power of 40,000 and 80,000.

Referring to FTIR analysis (Fig. 4.) a wide absorbtion band is the range of 2500-3750 Cm^{-1} signed to hetrogen stretch of Hydroxide and the band witht the range of 820 to 1300 Cm^{-1} is the revolution mode of Hydroxide.

Fig. 4. FTIR analysis for SnO2 nanoparticles prepared in 400 °C for 4 H

It is highly possible that a hetrogen stretch band of Sn-O-Sn is formed by hydroxide groups for nano particles in 600 Cm^{-1} band ,for Sn-o in 500 ,for Ca-O in 600 to 700 region for Cl-Ca-Cl in 3400 and for O-H in 3130 .

Making nanosensor using the produced SnO2 nano particles

Making the substrate and Gold electrodes on the substance

Firstly ,a mold from SPK steel was made to make Alomina substances (%0.96) with the dimentiones of 25.4 *4*0.7 mm .Then, the Alomina powder was mixed with glue powder(2 percent of the original weigth)and pored into the mold. The powder was molded and pressed under the pressure of 20 Kg/Cm^2 .for strengthening the substances, they were sintered in two stages in 1200 and 1700 C.

For making Gold electrodes on the substance and placing copper on the alumina ,the Spottering device was used and a layer of copper with the thickness of two microns was created .the circuit of heater and electrode was printed on the surface of the copper layer by lithography method.

After creation of the film , for cleaning the circuit from wax or other materials , the cleaning bath wes used and Acetone was used as the final activator in order to solve the oxide layer.

Using plating ,a gold layer is precipitated on the copper surface ,and the heater and the layer coated electrode is prepared for the next stage as can be seen in Fig. 5.

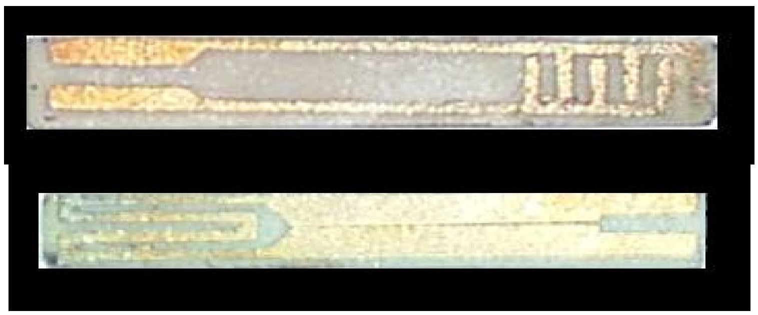


Fig. 5 . electrode and heater

Layer coating by SnO2 and attaching Gold conducting wires on the substance

For layer coating of Carbon monoxide sensitive materials on the electrode ,firstly 80 miligrame of SnO2 powder is grinded together with 6 miligrams of ethyl cellulose as the organic adhesive to make the completely fine. Then, about 30 micro litre of alpha terpinoil is added to the product by Ependorf as a solvent .by grinding again a complete uniform matrial is gained.

By placing the sensitive layer coated on the electrode in room temperature ,the ink covers the surface evenly and fills the boundaries of the network and evaporates the solvents which are volatile in room temperature.

In the next step, the system is put under IR Lamps and is posed to 150°C which eliminates the organic content and the layer is stabilized mechanically and attached stronger to the substance.After forming the SnO2 layer on the substance,for conducting electricity to heater,Gold wires were attached on the lower and upper Gold electrodes by Silver glue .After that,the whole system is placed in the chamber for tests in which the Carbone monoxide is injected into the chamber.

Sensor sensitivity and stability analysis

A voltage of 1.7 volts and current of 0.5 amper was used for heating substance to increase it's temperature to 200°C .referring to the prvious research on SnO2 Nano powder ,the maximum sensitivity is achieved in 200°C [4] .For measuring the temperature of the sample surface,a digital thermo couple was used .By conducting the other two Gold wires to the upper circuit(Signal Circuit) and by recording the changes in resistance of this wires by multimeter,the diagram of resistance versus the time for injections of Carbon monoxide is illustrated in Fig.6.

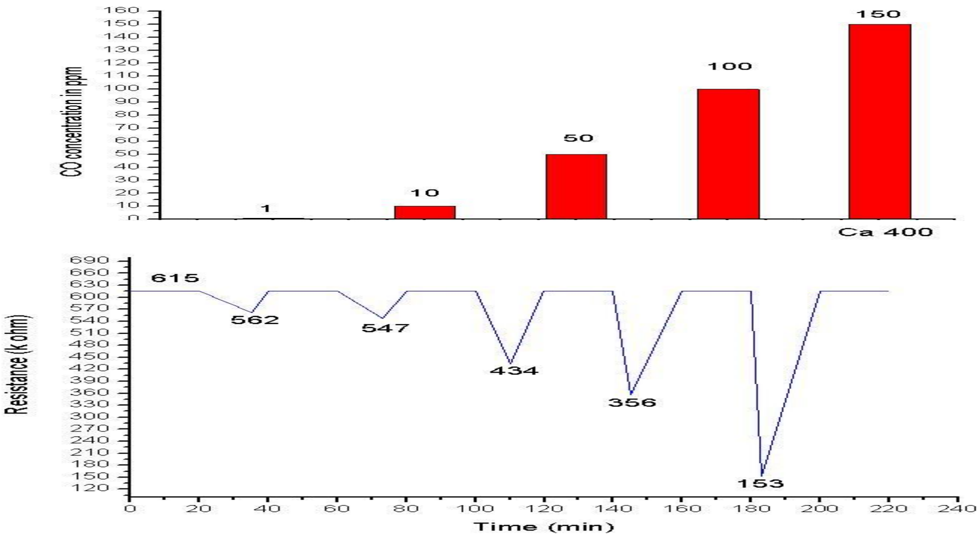


Fig.6. diagram of resistance versus the time for CO sensor (1-150 ppm)

Injecting Carbone monoxide gas with different concentrations to the chamber,the sensor shows different levels of sensitivity and the raise in the sensivity of the sensor with the increase of the amount of the gas can be seen in table 3 and Fig. 7.

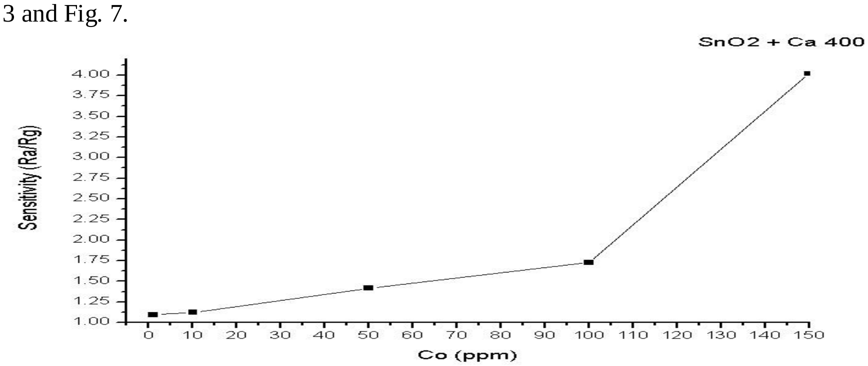


Fig. 7. diagram of sensitivity - concentration

Table 3. sensitivity – concentration for CO gas

Concentration (ppm)	**10**	**50**	**100**	**150**
Sensitivity (Ra/Rg)	1.09	1.12	1.41	4.02

Conclusion

The experiments show that the nano particles of SnO2 produced in the temperature of 400^{o}C ,with the average size of 7 nanometers are used in the sensor and act properly in all aspects of a good sensor(sensitivity and stability).By increasing the temperature ,the size of the nano particles increased .based on the thermography diagram, the temperature of 400C is the temperature in which the particles are stable .the good sensitivity of the sensor is due to the good porosity of the made nano particle .whith increase og the injected cabon monoxide ,the resistace of the the sonsors decreases considerably and its sensitivity raises incredibly.

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