



Trade Science Inc.

Materials Science

An Indian Journal

Short Communication

MSAIJ, 6(4), 2010 [234-236]

Synthesis of ZnTiO₃ and its gas sensing application

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Received: 30th May, 2010 ; Accepted: 9th June, 2010

ABSTRACT

Zinc-Titanate powder (ZnTiO₃) was synthesized by mechanochemical technique using TiO₂ and ZnO as a starting material in 1:1 mole ratio. The mixture were ball milled and heated between temperature 200^oc to 400^oc for various time. The ZnTiO₃ as a p-type semiconductor and it is H₂S gas sensor at 350^oc at concentration up to 100ppm. The XRD of this material shows crystalline nature, SEM indicate 38nm size, IR peak at 789cm⁻¹ and 977cm⁻¹ indicating formation of ZnTiO₃ material. © 2010 Trade Science Inc. - INDIA

KEYWORDS

H₂S gas sensor;
ZnTiO₃;
Wet chemical method.

INTRODUCTION

In recent year gas monitoring devices are demand for a rapidly growing range of application. Metal based chemical sensor have been used extensively for the detection of toxic pollutant gases such as NH₃, CO, H₂S, Cl₂ etc. The advantage of gas sensor is low prize, small size, high sensitivity and low power utilization. H₂S is the major pollutant gas in environment thus their there is a need develop a sensor for its detection. The H₂S is the toxic gas with threshold limit value 25 ppm for long term expose (6hr). There for gas sensor for detecting H₂S are required for environmental monitoring in chemical plant and research laboratories. Semiconductor oxide such as ZnO, TiO₂, SnO₂ have been required for detection of H₂S. The operation of this sensor based on reversible change in electrical conductance on expose of H₂S gas. TiO₂ is a semiconductor with a band gap energy E_g = 3,2 eV. If such material banded with another metal such as Zn then the gas sensing property increases than normal TiO₂. Thier are several method

for preparation of ZnTiO₃ compound such as solid state reaction, sol-gel reaction and mechanochemical method. In this work we have synthesized it by mechanochemical method because sol-gel technique is expensive. The prepared material tested for various gases on gas sensor and chrectorise by XRD, IR, SEM technique.

EXPERIMENTAL

Preparation of powder

The ZnTiO₃ powder prepared by mechanochemical method from 99.99% pure ZnO and TiO₂ powder as a starting material (Aldrich) The starting material taken in 1:1 mole ratio and mixed in mortar for 1 hr. Then this material calcinated for various temperature. At 200^oc for 2 hr, 400^oc for 2 hr, 600^oc for 1/2 hr, 800^oc for 1 hr and 1000^oc for 2 hr. The XRD of this material is taken for determining crystallographic structure with Cuk alpha radiation source. The FTIR technique identification of prepared material and SEM used to study surface morphology of the prepared material. The pre-

pared material were used for gas sensing application by using thick film. The thick film were prepared by screen printing technique. The 100 mg ethyl cellulose and 1 gm prepared material is mixed properly then paste is prepared by adding organic binder. The paste was screen printed. The prepared film fired at 550°C for ½ hr. The gas sensitivity of material is determine by

$$S = \frac{G_g - G_a}{G_a}$$

Where G_a –Resistance in presence of air.
 G_g –Resistance in presence of test gas.

RESULT AND DISCUSSION

Analysis of powder

The powder were characterise by XRD for crystalline structure (XRD Broker) using CuK alpha radiation source. The average grain size of powder were determined by Scherer formula. The average size of particle is 56 nm. In IR spectra of compound shows peak at 789 cm^{-1} due to Ti-O vibrational stretching also the peck at 977 cm^{-1} due to Zn-Ti stretching. The surface morphology of sample was determine by scanning

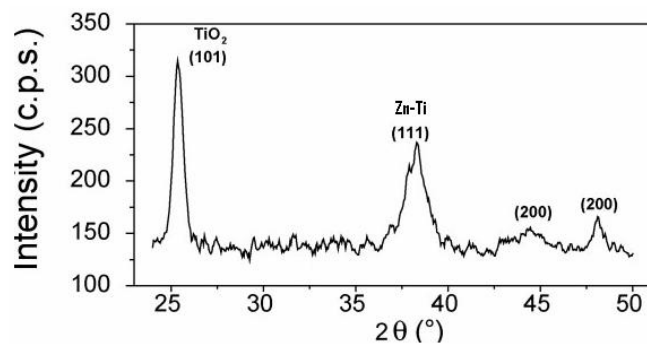


Figure 1 : XRD of ZnTiO_3

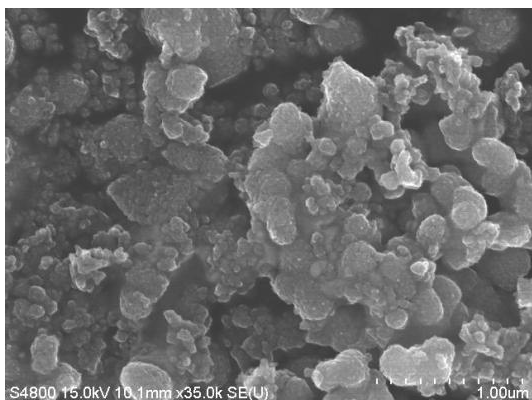


Figure 2 : SEM image of ZnTiO_3

electron microscopy (SEM). The size of grain determine by SEM was found to be 38 nm.

Gas sensing

The sensing performance of the film was observed by using static gas sensing unit. The heater plate was fixed on the base plate to heat the sample under set up to required temperature. The Cr-Al thermocouple was used to sense the operating temperature of the sensor. The output of thermocouple was connected to a digital temperature indicator. A gas inlet valve was fitted at one of the ports of the base plate. The required gas concentration inside the static system was achieved by injecting the known volume of a test gas using gas injecting syringe. A constant voltage was applied to the sensor, and current was measured by a digital Pico ammeter. Air was allowed to pass into every gas exposure cycle.

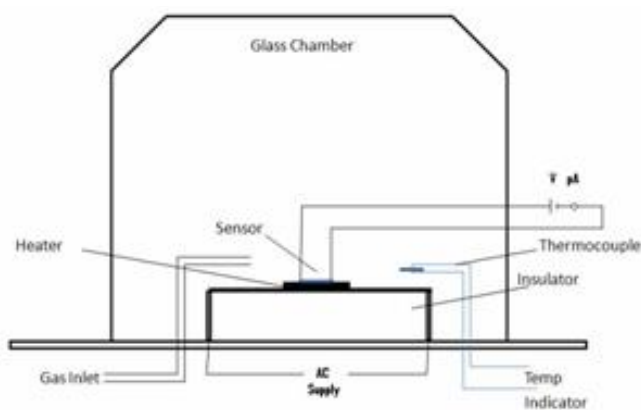
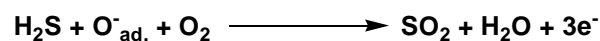


Figure 3 : Gas sensing unit.

Gas-sensing mechanism

The Zn-TiO_3 surface layer responsible for gas sensing property. There is a lot of adsorbed oxygen O_{ad} on the surface of Zn-TiO_3 material at room temperature. The chemical reaction on surface changes the the concentration of conduction electron that is responsible for gas sensing property. Initially there is large number of chemiabsorb oxygen at the grain boundaries of ZnTiO_3 . The loss of such adsorbed O_2 increases he free electron concentration of the surface and imparted the film conductance due to the increase in free electron. The reaction of H_2S gas in gas sensor as follows.



The above chemical reaction is for H_2S gas as comparison to other gases H_2S can easily donate the elec-

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tron by reacting it with adsorbed oxygen. After exposing the H₂S gas in sensor it react with adsorb oxygen and conductance get increases rapidly.

Gas sensing performance

Sensitivity with temperature

The bellow fig. shows variation of sensitivity for H₂S gas (100 ppm) with operating temperature ranging from 50^oc to 400^oc. It is noted from graph that response increases with increase in temperature. It is maximum at 350^oc and further decreases with decrease in temperature.

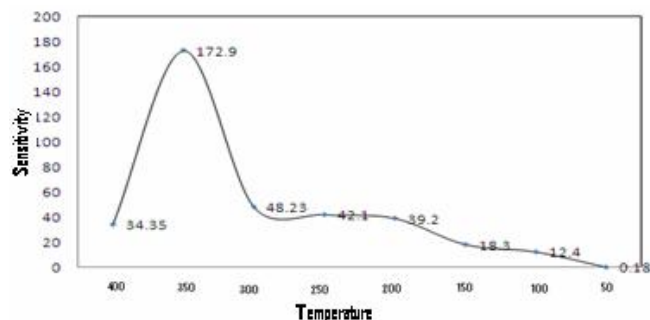


Figure 4 : Effect of temperature on sensitivity of ZnTiO₃

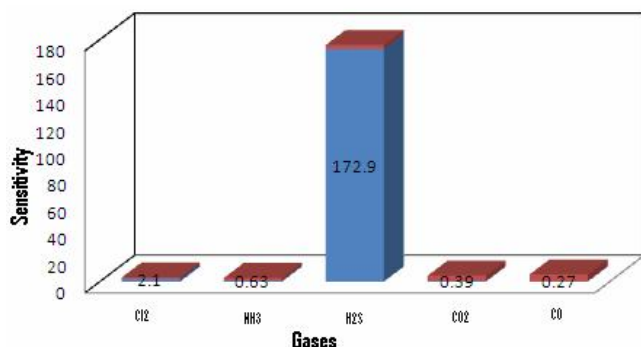


Figure 5 : Selectivity of ZnTiO₃ for H₂S gas with other gases at 350^oc.

CONCLUSION

In this study, the structures and sensing properties of ZnTiO₃ film as an H₂S gas sensor obtained by mechanochemical method. The structural characteristics shows that crystalline nature of ZnTiO₃. The SEM result shows that particle size in the range of 38 nm due such size the gas sensitivity get increase as compared to bulk zinc Titanate. The material shows good H₂S sensitivity up to 100ppm at 350^oc.

ACKNOWLEDGEMENTS

Authors are grateful thanks to Dr.V.B.Gaikwad, principal of K.T.H.M.College, Nasik to providing helpful guideline and instrumental facility. In this work. Author sincerely thank to UGC New Delhi for financial assistance to this research project. Author also thank to S.S. Gaikwad for his wholehearted co-operation in this work.

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