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DEPOSITION OF TITANIUM DIOXIDE (TiO2) THICK FILM FOR GAS SENSOR TECHNOLOGY

P. K. Saraswat, Ph. D.

Associate Professor, Deptt of Physics, Narain College Shikohabad (Firozabad) U.P. -283135

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Abstract

High-energy particles collide with the source target and knock out the atom or molecule from the source target. The ejected atoms or molecules condensed onto a substrate to form a thin film. . Materials to be deposited is converted from the condensed phase into vapor phase by physical means. The vapor transport from its source to the substrate. The vapor condenses on the substrate to form the thin films. Different techniques used for the characterization of sensing material's properties such as structural, morphological, optical and electronic are described. Near edge X-ray absorption fine structure spectroscopy is performed to understand the modifications in the electronic structure of fabricated film across the Fermi level. TiO2 thin film fabricated using the thermal evaporation method and characterized. The microstructural, optical properties and electrical properties of fabricated TiO2 film are studied using XRD, AFM and U-V absorption spectra. We found that TiO2 thin film shows the anatase crystalline phase and crystallite size of the film was ~ 60.77 nm and microstrain was ~ 0.0026. AFM measurement reveals that the average grain size and roughness parameter was~ 84 nm and~ 23.2 nm. The optical band gap was calculated from U-V spectra and it is~ 3.2 eV. We observed from XAS spectra that TiO2 film is anatase structure. The fabricated TiO2 thin films may be used in gas sensing devices.

Keywords: *Semiconductor, Photocatalytic, Semiconductor metal oxide, Spectroscopy.*

Material and Methods: The semiconductor metal oxide such as TiO2, ZnO has been used in present investigation as sensing materials. The undoped TiO2 and CdS-doped TiO2 sensing materials are prepared in the lab and studied their structural, morphological, optical, electronic and sensing properties for various hydrocarbon gases.

Properties Titanium Dioxide

Formula	TiO2
Density	4.20 g.cm^{-3}

Dielectric constant	4
Refractive Index	2.48 (Anatase), 2.58 (Rutile)
Energy band gap	3.05 eV (Rutile), 3.2 eV (Anatase)
Melting point	18430 C
Boiling point	29720 C
Solubility in water	Insoluble
Appearance	White Solid

Applications

The most important function of titanium dioxide, however, is in powder form as a pigment for providing whiteness and opacity to products as paints, coatings, plastics, fiber food, and cosmetics. Titanium dioxide is the most widely used white pigment and has a very high refractive index-surpassed only by diamond. Even in mildly atmospheres titanium tends to lose oxygen and become substoichiometric. In this form, the material becomes a semiconductor and the electrical resistivity of the material can be correlated to the oxygen content of the atmosphere to which it is exposed. This electronic property including photocatalytic, optical properties of TiO2 has been widely used as gas sensors in the class of semiconducting oxides. Titanium dioxide is one of the most favoured compounds for nanostructures semiconductor gas sensors because it is non-toxic, inexpensive, highly photoactive and easily synthesized and handled. Titania has been used as photocatalyst under UV light, pigment in cosmetic and skincare products, in wastewater remediation, etc.

Sensor Parameters

To characterize the features like performance, quality, and accuracy of fabricated sensors, we need some standard parameters as a response, selectivity, sensitivity, response recovery time, reproducibility, etc. These parameters are used by several workers but sometimes the standard definitions may deviate.

Response

The 'percentage change in measuring parameter' to a particular gas concentration is defined as '*Response*' parameter ***S*** of the sensor as $S = 100\ (Ra- Rg)/\ Ra$; where, Ra = measuring parameter of the sensor in air, Rg = measuring parameter of the sensor in the presence of gas exposed. The Measuring parameter R may be resistance, conductance or capacitance depending upon the technology employed in the fabrication of device viz. thick film, thin film or MOSFET, etc.

Sensitivity

The word 'Sensitivity' is defined as the slope of the linear region of the plots traced as 'Response vs. Concentration' curve for a particular vapor and is given by **Sens= $\Delta P/\Delta C$**

where ΔP = change in response, ΔC= change in concentration.

Fabrication of Titanium dioxide Thin Film Gas Sensor using Physical Vapor Deposition Method:

Vacuum Chamber

The chamber is fabricated from electrochemically polished stainless steel. Three circular glass windows enable visual inspection of the coating process. When the chamber is placed on the base plate it makes vacuum-tight seal with the base plate by mean of an L type neoprene gasket. A cooling water pipeline is coiled on the outer wall of the chamber to prevent overheating and to reduce the out gassing by circulating the water.

Vacuum Pumping System

The pumping system included rotary pump model ED-15 (pumping speed of 250 lpm) diffusion pump model OD-114 (pumping speed of 280 lpm) as a high vacuum pump along with combination valve model CV-25 and High vacuum valve model QSV-6 roughing backing valve.

Procedure for Fabrication of Thin Film

The thin film technology has the flexibility of changing fabricated device properties by varying shape and size of components allowed by the thermal evaporation method and the choice of substrate. We fabricated TiO2 thin film using Physical Vapor Deposition (PVD) method. PVD is a conventional process for thin-film deposition. The PVD process involves at least the following steps:

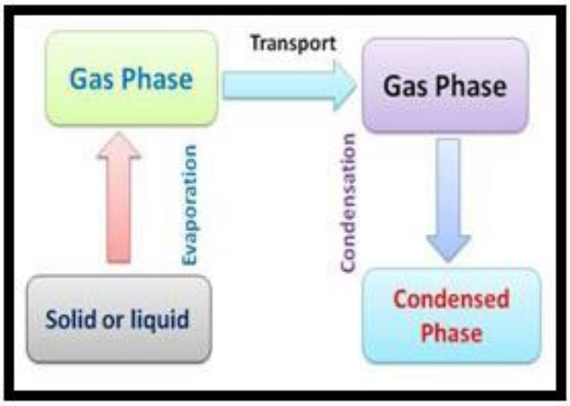
1. Materials to be deposited is converted from the condensed phase into vapor phase by physical means.

2. The vapor transport from its source to the substrate.

3. The vapor condenses on the substrate to form the thin films.

There are several ways to resolve the key step for the PVD process, that is, to convert the solid phase into the gas phase. In general, three major processes have been utilized: **(1).** The thermal evaporation method, **(2).** the sputtering growth method and **(3).** Pulse laser deposition.

For the thermal evaporation method, the source materials are placed into crucible, and the crucible can be heated either by resistance or by an electron beam to its melting temperature so that there is enough vapor coming from the source that can be deposited onto the substrate.



Schematics illustrating the general step and physical mechanism for PVD process

Methodology used in preparation of sensing materials and sensors - For the sputtering growth, a source-target is used, and inside the vacuum chamber high-energy ions are generated by ionization. Those high-energy particles collide with the source target and knock out the atom or molecule from the source target. The ejected atoms or molecules condensed onto a substrate to form a thin film.

Different steps used in the deposition of TiO2 thin films

I. First, the creation of materials (TiO2) to be deposited in an atomic, molecular or particulate form before to the deposition. II. The different methods such as physical methods or chemical methods are used to transport materials to the substrate in the form of vapor stream or solid or spray etc. III. On the substrate deposition of materials and the film growth by the nucleation growth process.

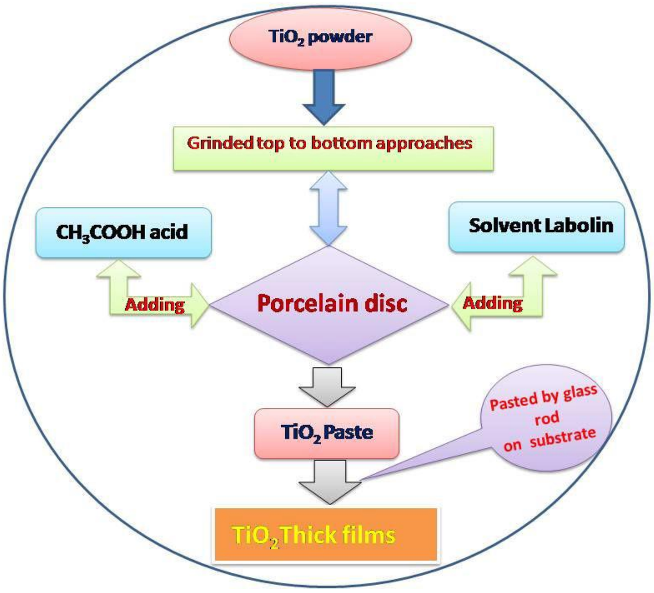
Deposition of Titanium dioxide Thick Film

The basic steps in thick-film technology require; development of formulated paste, pattern generation, printing and firing. In the thick film fabrication process, a vital role is acted by sensing paste consists of a functional component (metal, semiconductor, dielectric) and a solvent (labolene). The apparatus used for the fabrication of the sensor during the course of investigation is listed as: Powder grinder (Porcelain Disc), glass rod, solvent, acetic acid, tissue paper, a clean glass plate or alumina substrate.

Procedure of making TiO2 Thick Film

Initially, we take some amount of TiO2 powder in porcelain disc and grinded manually top to bottom approaches for 1hours to 2 hours. The grinding process is complete after suspended some amount (~ 5-10 ml) of CH3COOH acid in the porcelain disc. Further, TiO2 paste is obtained by adding ~ 8-10 drops of labolene solvent. The TiO2 pastes are pasted on a glass substrate or alumina substrate. The fabricated samples are annealed at 573 K for getting better adherence of the films. Different steps for the fabrication of undoped and CdS-doped TiO2 samples.

Flow chart for the fabrication of TiO2 thick film



Experimental Setup used for the Measurement of Sensor Response

For the measurement of the response of the fabricated sensor a test chamber has been made in which provisions are made for electrical connection and inlet/outlet for test gases. The volume of the test chamber is 2 litter (2047 ml exactly). The connectivity for voltage supply and resistance measurement is available through insulated gaskets on the base of the chamber. The resistance measurement of the fabricated sensor in air and gas environment is carried out with the help of Dual DC power supply (LD3202) and Digital Multimeter (Aplab 107N). The responses of the fabricated sensor are measured in air ambient at room temperature with varying test gas concentrations.

In nature, the bulk form of TiO2 is existing in one of three basic crystal structures: rutile, anatase (both are tetragonal) and brookite (orthorhombic). Anatase and brookite are the well-know metastable phases and convert irreversibly to rutile phase at the temperature range of 400-800 0C depending on various conditions. The most widely used forms of TiO2 as gas sensing materials are tetragonal anatase.

The lattice parameters of anatase are a = b = 0.376 nm, c = 0.948 nm, the rutile are a = b = 0.459 nm, c = 0.296 nm. Each titanium atom is connected with 6 oxygen atoms and form TiO6 octahedron in both forms. Through both the anatase and rutile phases belong to orthogonal crystal system, the distorted degree of each TiO6 octahedron presents obvious difference. The symmetry of anatase phase is poor as it possesses severe distortion, while rutile phase has near-perfect orthogonal crystal system with small distortion. Such differences in the structure are sufficient to result in different band structures, electronic and optical properties, and thus further influence their performance in many applications including gas sensing. One of the most important fundamental properties is the band structure of TiO2 which plays a significant role in electronic and optical properties that embodied in charge carrier mobility, redox potential, and light absorbance and so on. Presently, most studies are focused on anatase because of its higher reactivity and simpler structure as compared to the rutile TiO2. The value of band gap for bulk anatase and rutile TiO2 are 3.2 eV and 3.0 eV respectively. The doping with metal or nonmetal elements can affect the position of conduction band (CB) and valance band (VB) or form a new energy level in the band gap thus changing the band structure of TiO2.

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