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Characterization of Advanced Spray Pyrolysis Technique Based Deposited Titanium Dioxide Thin Film at 550°C Temperature

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Abstract- Titanium dioxide (TiO2) thin films have more interest due to the wide scope for the development of novel solutions in the field of healthcare and sensing electronics. TiO2 is considered as an inert and safe material and has been used in many applications for decades. It has a wide range of industrial, technological applications and an important compound suitable for fuel cells, solar cells, and sensors. Physical properties of TiO2 are depended on the phase structure, pores, and particle size. High refractive index, chemical stability, and poor solubility are properties that add to its practical applicability. A naturally occurring oxide has a wide range of applications. Thin films with high transparency in the visible range of the electromagnetic spectrum have been deposited. Titanium isopropoxide resulting powders is characterized by thermo-gravimetric analysis (TGA) and thermal differential analysis (DTA). Advanced spray deposited titanium dioxide thin film is prepared at 550^0C have been characterized by X-ray diffraction.

Keywords- *Spray Pyrolysis, Diffraction, Pores and Titanium Dioxide*

I. Introduction

Thin film techniques played an important role for sensor manufacturing industry widely. Manufactured sensors are playing important role in gas sensing technology. Due to the importance titanium dioxide, in this research spray pyrolysis method are used for the development TiO_2 thin film.

II. Spray Pyrolysis Methodloogy

Because of simplicity and better productive performance of thin-film, the spray pyrolysis technique has more demand. It is one of the most well-known chemical techniques to deposit various types of materials in the form of thin films, well in productivity from an uncomplicated apparatus.

It has more advantages; It is well known to dope thin films in any amount or proportion. It helps to low as well as high-quality substrates. With the help of the spray pyrolysis method thickness of the films and deposition rate can be controlled. Spray pyrolysis technique helps to produce films on less robust materials. There is no restriction on dimension, substrate and surface profile. Spray pyrolysis produces layered films with a variety of thicknesses. It gives well-characterized film surfaces, provides compact films, uniform and that no side effects from the substrate occur. The enhancement in deposition efficiency and improvement in the quality of the thin films can be achieved with these techniques.

III. Experimental Set Up

Before Chemical deposition of TiO2 films using advanced spray pyrolysis method includes the substrate cleaning, solution preparation, and deposition. The optimized preparative parameters were used to deposit the nano-crystalline and feasible TiO2 films for gas sensing applications. Advanced spray deposited TiO2 thin films are characterized by X-ray diffraction for structural characteristics. To carry out uniform and crack-free films, sufficient cleaning of the substrate and proper surface treatment prior to the deposition is essential, as the contaminated surface provides nucleation sites facilitating growth resulting in non-uniform films with different orientations and impurities. Mainly, defect formation and density always depend on the cleanliness of the production environment so the effective surface cleaning method should be chosen as per particular substrate.

The glass micro-slides of dimensions 75×25×1.35mm have been used as the substrates. The step by step procedure is adopted for substrate cleaning. slides were washed with distilled water, boiled with chromic acid for 30 minutes and substrate washed by double distilled water. The substrate is cleaned by ultrasonically and exposed with methanol vapors for 4-5 minutes and finally, the substrates will be ready for deposition.

IV. Analysis of Titanium Isopropoxide

To analyses, the range of core temperature (chamber temperature), transformation phase and mass loss events which is to occur during heating of titanium isopropoxide

$[C_{12}H_{28}O_4Ti]$ powder was investigated by simultaneous thermo-gravimetric (TGA) and differential thermal analyzer (DGA).

In the beginning, in step I (300-360 K), 12% weight loss of titanium isopropoxide was traced. This weight loss is exposed by the endothermic peak at 560 K attributable to the evaporation of physiorbed water from the precursor. Subsequently, a slow decay of TGA curve was observed in Step II (380-430K). Further, in Step III, a rapid decay in TGA curve has occurred during 420-520K. Consequently, the total weight loss of 55% was observed during step II & III. These resultant weight losses are caused by the decay leading to the decomposition of acetate groups. A weight-loss around the temperature of 600 K corresponds to an exothermic peak signifying the TiO2 phase formation and becomes stable after ~ 600 K. After 600 K, the TGA trace remains stable with low weight loss; which indicates the complete decomposition of titanium isopropoxide to form a stable TiO2 phase. The exothermic peak in DTA plot suggests phase formation [6,8].

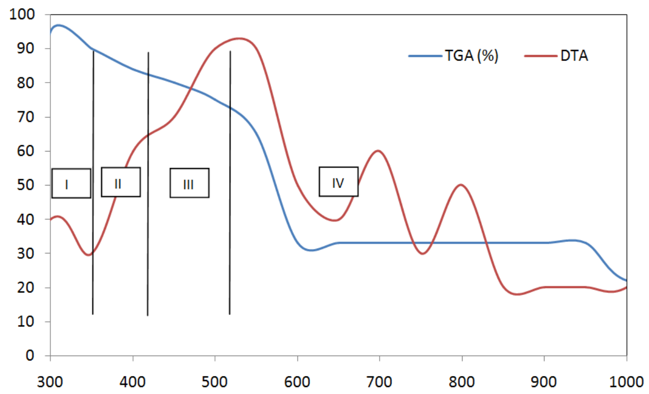

Figure 1. Analyzing behavior of Titanium isopropoxide

V. DEPOSITION OF TITANIUM DIOXIDE THIN FILMS

Un-doped TiO_2 thin film synthesized by advanced spray pyrolysis technique. The reaction chamber, substrate heater, temperature control system, and nozzle assignment are the main part of the advanced spray pyrolysis system. A pre-optimized 0.1M non-aqueous solution of high purity titanium isopropoxide (Thomas Baker, India) was prepared in ethanol or ethylene glycol $[C_2H_5OH]$ to obtain TiO_2 films. The chemical solution was atomized through a glass nozzle and the reactants decomposed at the reaction chamber pyrolytically and oxide particles thrown towards the glass substrates and deposited. At the time of course of the deposition, the nozzle to substrate distance is kept 40 cm and compressed air at a pressure of 15LPM was used to control the solution spray rate at 6 ml/min. For the study of the effect of core temperature on the TiO_2 film properties, films were deposited onto glass substrates kept at constant 710 K substrate temperature; by varying the core temperature from 710 to 840K at the step of 50 K.

Air pressure: 15 LPM, spray rate: 6 ml/min, nozzle to substrate distance: 40cm, reaction chamber temperature (700K) constant, the chemical solution was atomized through a glass nozzle. The resultant aerosols arrive in the reaction chamber and lead to oxidation of the ingredient. The fine particles of the pyrolytic reaction product pushed up and reach to preheated substrates kept at 710 to 840K temperatures.

Again, to investigate the film thickness effect, different volumes of spraying solutions were atomized by compressed air onto preheated glass substrates maintained at 710K to obtain films with different thicknesses. At the time deposition, other parameters viz. nozzle to substrate distance (40cm), core temperature (900K), spray rate (6ml/min), air pressure (15LPM) were essentially kept constant. Throughout the above experimentations, both the substrate and core temperatures were controlled using electronic temperature controllers.

VI. RESULTS AND CONCLUSSION

The solution of titanium isopropoxide stirred with ethanol sprayed and enters the reaction chamber and pyrolytic decomposition takes place the results into titanium oxide particles. Titanium dioxide particles were thrown towards the upward direction and were deposited on the substrate which is kept above the reaction chamber [7].

A. X-ray diffraction characteristics

Advanced spray doped titanium dioxide thin film has been studied with X-ray diffraction. X-rd patterns of films grown at different core temperatures are given in figure 2. It is evident from the figure that the crystallographic properties of the TiO2 films largely depend on core temperature. Films are traced out and found amorphous in nature grown at low core temperatures with acetate traces, but films are found crystalline nature without acetate traces at higher core temperatures. A few weak peaks are observed for films grown at 710K and 840K core temperatures and are difficult to discriminate from the background noise. Conversely, well-defined (101) peak along with reflections from (004), (201) and (203) diffraction planes are clearly observed for the film obtained at 900K core temperature [9]. This indicates the formation of polycrystalline TiO2 [JCPDS No. 21- 1272]. The films deposited at 900K are dominantly oriented along (101) plane. With a further increase in core temperature, preferential growth slightly decreases.

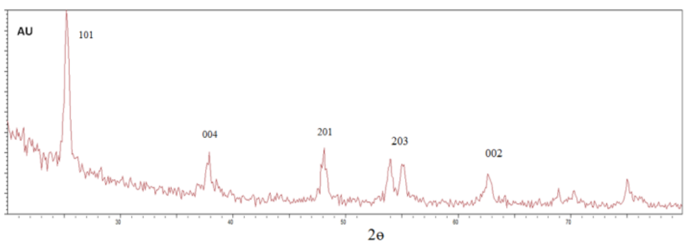

Figure 2. X-ray diffraction of TiO_2 thin film at 550°C temprature

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