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TiO_2 Thin Films: A Review

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***Abstract* - Oxides of materials have huge applications in near about all developments and technologies. The proper synthesis of oxides with different doping, it is widely used for various purposes. In this review article, synthesis of TiO_2 by sol gel method and different methods for deposition of thin films is explained because; sol gel method is found convenient, suitable, simple, low-cost method.**

Review said that Titanium dioxide (TiO_2) is found the vast applicable material. It has lots of application. TiO_2 itself or with doping with proper material it used as a photo sensor, humidity sensor, ethanol sensor, organic vapour sensor, environmental chemical species sensor, pH sensor etc. The different properties of TiO_2 are studied and explained in this review article.

***Index Terms* - Synthesis, Deposition, Thin Films, Sol - gel, spin coating, Sensing, Dopant, etc.**

1.INTRODUCTION

Since ancient time, our ancestor has started the journey of research. The latest technology in science is nothing but the need of human being. As per the need, we developed by using the materials on the Earth in proper manner and proper proportion. We use all materials in bulk quantity, but before the use we must know the properties of smallest unit. To know the property, structure etc of smallest form of material on their nano scale we must know the different thin or thick film deposition techniques. There are various method of thin film depositions as well as synthesis of material used by the researchers. Researchers are busy to find the different applications of the materials. With proper synthesis method and with proper doping of materials, different materials have the huge applications in sensing area. Most of the materials are used as a photo sensor, gas sensor, humidity sensor, chemical sensor, temperature sensor etc. [1]

Thin films can be made by using, sterilized substrate, base material which is to be deposit, a source for the film material, needs to stick material with substrate and etching of film to attain the certain pattern of the film.

Many oxides of materials found on earth are widely used in different sensing devices. TiO_2, SnO_2, ZnO, ZrO_2, CeO_2-ZrO_2, Cr_2O_3, WO_3, Al_2O_3, Y_2O_3 etc are the most important oxides used as sensors. [2-5]

Out of all the oxides present on the earth, Titanium oxide is found to be widely used material and the study related to TiO_2 is going on large scale by the researchers. Titanium dioxide (TiO_2) has been an important material for decades, combining numerous attractive properties in terms of economy (low price, large availability) or ecology (non-toxic), as well as broad physical and chemical possibilities.

It is found that, TiO_2 is used as a photo sensor, humidity sensor, ethanol sensor, organic vapor sensor, environmental chemical species sensor, pH sensor etc.

2. APPLICATIONS OF TiO_2

When pure polypyrrole (PPy) and TiO_2 nanoparticles /polypyrrole (TiO_2 NPs/PPy) form composite thin films on an alumina substrate, characterization shows that, it can be used as humidity sensor. [6]

The sensor made of TiO_2 NPs/PPy composite thin films, using the added amount of TiO_2 NPs as 0.0012 g showed the highest sensitivity, smaller hysteresis and best linearity. Moreover, other sensing properties, such as effects of applied frequency, ambient temperature [7], response and recovery time and long-term stability were also investigated [8].

A new type of solar cell (called of the third generation) based on the use of a TiO_2 layer sensitized with a dye (dye-sensitized solar cell [DSSC]). This cell differs from the massive (single crystal or polycrystalline) silicon cells based on a p-n junction. [9,10,11]

TiO_2 nanotubes are typically one-dimensional material, which has a wealth of physical and chemical properties and low production cost, and therefore, it bears a broad application prospect In particular, recent studies show that, due to large specific surface area and nanosize effect, compared with other forms of nanostructures, TiO_2 nanotubes show great potential for development in photo catalysis sensors, solar cells and other areas. [12]

Undoped TiO_2, chromium-doped TiO_2:Cr and TiO_2-SnO_2 synthesized by flame spray synthesis (FSS) technique acts as a hydrogen sensing (Gas sensor) [13]

TiO_2 has excellent physical and chemical properties. It is low price, large available, non-toxic material. The different properties of TiO_2 like structural, electrical, optical etc can be obtained by synthesis parameters, deposition conditions and dopands. [14]

3. DEPOSITION OF TiO_2 THIN FILMS

There are various methods of decomposition of TiO_2 films. The widely used techniques are -

Spin coating
Sol-gel technique
Chemical bath deposition
Spray pyrolysis technique
Electroplating technique
Dip coating

3.1 Spin coating:

It is a procedure used to deposit uniform thin films onto flat substrates. Usually a small amount of coating material is applied on the center of the substrate, which is either spinning at low speed or not spinning at all. The substrate is then rotated at speed up to 10,000 rpm to spread the coating material by centrifugal force. A machine used for spin coating is called a spin coater [7,15,16]

3.2 Sol-gel technique:

It is a wet-chemical process that involves the formation of an inorganic colloidal suspension (sol) and gelation of the sol in a continuous liquid phase (gel) to form a three-dimensional network structure. [2,5,9,15,16]

3.3 Chemical Bath deposition:

Chemical Solution Deposition (CSD) comprises all solution based thin- film deposition techniques, which involve chemical reactions of precursors during the formation of the oxide films, i. e. sol-gel type routes, metallo-organic decomposition routes, hybrid routes, etc. [17]

3.4 Spray pyrolysis technique:

It is a process in which a thin film is deposited by spraying a solution on a heated surface, where the constituents react to form a chemical compound. The chemical reactants are selected such that the products other than the desired compound are volatile at the temperature of deposition. [15,16,18]

3.5 Electroplating:

Electroplating is a general name for processes that create a metal coating on a solid substrate through the reduction of cations of that metal by means of a direct electric current. The part to be coated acts as the cathode of an electrolytic cell, the electrolyte is a solution of a salt of the metal to be coated; and the anode is usually either a block of that metal, or of some inert conductive material [12]

3.6 Dip Coating:

Dip coating is one of the most effective processes for the production of PSC. The solution substrate is immersed in the solution for effective formation of the material. Once the material is deposited then the substrate can be removed by evaporation which will result the thickness of the layer are unique. The major forces that are used for the dip coating process are force of inertia, viscous drag, gravitational force, and surface tension. The merits of dip coating are low cost and layer thickness can be easily adjusted. The drawbacks of dip coating are process is slow and it has the ability to block the screen, which will create major impact in the final product. [18-24]

All above methods have their own advantages and different conditions for their operations. Out of all above methods, sol gel method and spin coating are found most convenient, suitable, simple, low-cost technique.

4. DETAILS OF SOL GEL METHOD

The sol-gel process that leads to the formation of TiO2 films is based on mechanisms of hydrolysis and polycondensation of titanium alkoxides mixed with alcohol and catalytic agents. There are various kinds of Ti alkoxides such as titanium isopropoxide (Ti(OiPr)4) and titanium ethoxide (Ti4(OEt)16), among others, that need to be used preferentially with their correspondent alcohol. The precursor solution, also called sol, is a colloidal suspension of Ti surrounded by ligands, with physical-chemical properties adequate to the formation of a film. After a deposition, which can be by dip-coating, spin-coating, and spray-coating processes, the film is formed by a wet gel that became a dry gel after drying process. The hydrolysis of the alkoxide group to form Ti─OH occurs due to nucleophilic substitution of O─R groups (alkyl group) by hydroxyl groups (─OH) and the condensation of the group Ti─OH, to produce Ti─O─Ti and by-products (H2O and ROH).

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$$Ti(OR)n+nH_2O \rightarrow Ti(OH)n+nROHTiORn+nH_2O \rightarrow TiOHn+nROH \quad e1$$

$$Ti(OH)n \rightarrow Ti(On/2)+n/2H2OTiOHn \rightarrow TiOn/2+n/2H2O \quad e2$$

$$TiOR+TiOH \rightarrow TiO_2+ROHTiOR+TiOH \rightarrow TiO_2+ROH \quad e3$$

This mechanism is relatively complex because the reactions occur simultaneously during the process of deposition. In this proposed mechanism, the alkoxides precursor passes by the sequences, oligomer, polymer, and colloid, and it finishes as an amorphous porous solid structure. Thermal treatments are used for the preparation of nanocrystalline thin films. With the use of doping salts in the precursor solutions, the mechanism becomes more complex due to the introduction of other metals in the gel network.

The dip-coating technique consists into dip a substrate in the sol and removes it at constant speed, resulting in an M─O─M oxide network that forms a wet gel film. The network structure, the morphology, and the thickness of the film depend on the contributions of the reactions of hydrolysis and condensation that must occur in approximately the same velocity of substrate withdrawal. Otherwise, the solution may run down the substrate. These properties may be controlled varying the experimental conditions: type of organic binder, the molecular structure of the precursor, water/alkoxides ratio, type of catalyst and solvent, withdrawal speed, and solution viscosity. After the deposition, the gel film is formed by a solid structure impregnated with the solvent, and a drying process can be used to convert the wet gel in a dry porous film. Denser film can be tailored by different temperatures of thermal treatment, leading to films with different specific surface areas and porosities. This mechanism is relatively complex because the reactions occur simultaneously during the process of deposition. In this proposed mechanism, the alkoxides precursor passes by the sequences, oligomer, polymer, and colloid, and it finishes as an amorphous porous solid structure. Thermal treatments are used for the preparation of nanocrystalline thin films. With the use of doping salts in the precursor solutions, the mechanism becomes more complex due to the introduction of other metals in the gel network.

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Denser film can be tailored by different temperatures of thermal treatment, leading to films with different specific surface areas and porosities.

5. DETAILS OF SPIN COATING METHOD

Spin coating generally involves the application of a thin film (a few nm to a few um) evenly across the surface of a substrate by coating (casting) a solution of the desired material in a solvent (an "ink") while it is rotating. Put simply, a liquid solution is deposited onto a spinning substrate in order to produce a thin film of solid material, such as a polymer.

The rotation of the substrate at high speed (usually >10 rotations per second = 600 rpm) means that the centripetal force combined with the surface tension of the solution pulls the liquid coating into an even covering. During this time the solvent then evaporates to leave the desired material on the substrate in an even covering. [7, 24-26]

This process can be broadly divided into 4 main steps:

1. Deposition
2. Spin up.
3. Spin off
4. Evaporation

In the initial step, the solution is cast onto the substrate, typically using a pipette. Whether the substrate is already spinning (dynamic spin coating) or is spun after deposition (static spin coating), the centrifugal motion will spread the solution across the substrate.

The substrate then reaches the desired rotation speed – either immediately or following a lower-speed spreading step. At this stage, most of the solution is expelled from the substrate. Initially, the fluid may be spinning at a different rate than the substrate, but eventually the rotation speeds will match up when drag balances rotational accelerations – leading to the fluid becoming level.

The fluid now begins to thin, as it is dominated by viscous forces. As the fluid is flung off, often the film will change colour due to interference effects. When the colour stops changing, this will indicate that the film is mostly dry. Edge effects are sometimes seen because the fluid must form droplets at the edge to be thrown off.

Finally, fluid outflow stops and thinning is dominated by evaporation of the solvent. The rate of solvent evaporation will depend the solvent volatility, vapour pressure, and ambient conditions. Non-uniformities in evaporation rate, such at the edge of a substrate, will cause corresponding non-uniformities in the film.

Advantages and Disadvantages of Spin Coating

The main advantages of spin coating are:

- The simplicity and relative ease with which a process can be set up coupled with the thin and uniform coating that can be achieved at various thicknesses makes it ideal for both research and rapid prototyping.
- The ability to have high spin speeds leads to fast drying times (due to the high airflow) which in turn results in high consistency at both macroscopic and nano length scales, and often removes the need for post-deposition heat treatment.
- Spin coating is a very low-cost way to batch process individual substrates compared to other methods, many of which require both more expensive equipment and high energy processes.

6. DETAILS OF DIP COATING METHOD

Dip coating refers to the immersing of a substrate into a tank containing coating material, removing the piece from the tank, and allowing it to drain. The coated piece can then be dried by force-drying or baking. It is a popular way of creating thin film coated materials along with the spin coating procedure. [18,24]

Stages of Dip Coating

The dip coating process can be, generally, separated into 3 stages:

- Immersion: the substrate is immersed in the solution of the coating material at a constant speed preferably judder free
- Dwell time: the substrate remains fully immersed and motionless to allow for the coating material to apply itself to the substrate
- Withdrawal: the substrate is withdrawn, again at a constant speed to avoid any judders. The faster the substrate is withdrawn from the tank the thicker the coating material that will be applied to the board.

Advantages:

Owing to its simplicity, this method lends itself to automation. Film thickness is controlled by coating viscosity and rate of withdrawal from the tank. Dip tanks come in all shapes and are sized to accommodate the largest object to be coated.

7. CHARACTERIZATION OF TiO_2

The characterization X-ray diffraction (XRD), specific surface area (BET), scanning electron microscopy observation (SEM), X-ray analysis (EDX), visible–ultraviolet diffuse reflectance spectroscopy (DRS) and Fourier transform infrared spectroscopy (FTIR) monitoring of surface acidity of TiO_2. The characterization results indicate an increase of TiO_2 anatase phase with the number of impregnations. The porosity and the surface area of all the photo catalysts prepared by supporting TiO_2 (anatase) on TiO_2 (rutile) were always higher [32,35]. TiO_2 (anatase)-based photo catalyst powders containing up to 20 mol% calcium, strontium or barium ions were prepared from a-titanic acid by calcining gels prepared from tri-ethanolamine-based sols at 600 ³C. The powders were characterised using X-ray diffraction (XRD), scanning electron microscopy (SEM), diffuse reflectance ultraviolet spectrophotometry, nitrogen sorption porosimetry and in situ infrared spectroscopy to examine surface adsorbed species. Compositions containing greater than 15 mol% alkaline earth ions resulted in largely amorphous materials. The residual anatase showed decreased crystallite sizes and increased crystallographic cell volumes with increasing concentration of alkaline earth ions, while the BET surface areas of the materials increased from around 80 m2 g21 (no additive) to 160 m2 g-1 at higher levels of additive. [33]

8. FUTURE WORK AND PREPARATION

According to review regarding Titanium dioxide (TiO_2) is found that, it is a vast applicable material. It has lots of application in the various fields. TiO_2 itself or with doping with proper material is used as a photo sensor, humidity sensor, ethanol sensor, organic vapor sensor, environmental chemical species sensor, pH sensor etc. A lot of work related to synthesis of TiO_2 and TiO_2 with doping already have been done and is going on. But study of TiO_2 in many fields, by different manner and by using a proper concentration doping is still challenge for new researcher.

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