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Preliminary Study Flourine Tin Oxide (FTO) Using Sol-Gel Spin Coating Techniques

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Abstract*:Flourine doped tin oxide is one promising material as Transparent Conducting Oxide (TCO) to replace the expensive FTO in solar cell electroda, as gas sensor, optical coating, and variable resistor. In this research, Flourine doped tin oxide (FTO) thin films on glass substrate were prepared by the sol-gel spin coating. The starting material for synthesis FTO material is Tin (II) Chloride dehydrate ($SnCl_2.2H_2O$), Hydroflouride (HF), and ethanol. As the material sol, Tin (II) Chloride dehydrate dissolved in ethanol, heated and stirrer using hot plate at 80 ℃ and 300 rpm for one hour. Then the solution using spin coating technique for one minute with 2000 rpm on glass substrate. The deposited films were baked at 150 ℃ for 10 minutes on oven at 100℃ and annealed at 650 ℃ for one hour in a tube furnace.*

Keywords: *FTO, sol-gel technique, substrate.*

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I. Introduction

The development of thin layer technology is already widely used in everyday life of which are used in sensors, solar cells, batteries and touch screen, one of the ingredients to create a thin layer is a compound Tin Oxide (SnO_2).A thin layer of SnO_2 is a type of n-type semiconductor, which has a chemical stability high, possess good transparency to light (the energy gap ~ 3.6 eV) [1], low electrical resistance, and has chemical stability high. Excellence a thin layer of SnO_2 widely applied in manufacturing industries in the transparent conductive electrode (TCO), solar cells, optical and electrical equipment gas sensor. Application TCO develops rapidly and has been applied to the devices of electronics such as LCD TVs, Plasma TVs, organic electroluminescence (EL), for example touch screen monitors on automatic tellermachine (ATM), ticket vending machines were installed in train stations, car navigation systems, handheld game consoles, mobile phones, and electrodes in solar cells [2].The SnO_2 attractive for development because the price is cheap compared to other semiconductors, nature responsive to a number of gases, has a long service life, and requires only a simple electronic device in the sensory implementation. To improve the performance of SnO_2 thin film material, research continues to be done to get the expected material characteristics. The SnO_2 often produced in pure form or doped with other materials.Based on the research that has been done, SnO_2 usually doped with indium or called ITO (Indium Tin Oxide) [3], ATO (Antimony Tin Oxide) [4], FTO (Fluorine Tin Oxide), and AFTO (Antimony and Fluorine-doped Tin Oxide) [5].In this research used doping SnO_2 Fluorine is an element in the capture of 40% HF compound. Fluorine is the show all the forms of the elements (ionized, nonionized) which means it is a chemical element that is very electronegative than other chemical elements.

The SnO_2 and doping concentration of HF use dilution with a ratio of (1: 99)%, (5: 95)%, and (10 : 90)% to be placed on the glass substrates using Sol Gel Spin Coating. Various methodsof growinga thin layer thathas been used before, such asRFSputtering[6], ChemicalsolutionDeposition[7], UltrasonicSprayPyrolysis[8], CathodicVacumArcDeposition[9], PhysicalVapor Deposition[10], andsol-gel [11]. However amongthese methods, sol-gel methodswidely used, because ofits low cost,its compositionis homogeneous, do notusea vacuumchamberwitha highlevel, the layer thicknesscan be controlled, andmicrostructureare quitegood[12].

Pure or doped SnO_2 thin films have been produced through a variety of different techniques, such as chemical vapor deposition, aerosol pyrolysis, sputtering, physical vapor deposition, laser abration, dip coating and sol-gel spin coating. All of these techniques have their respective advantages and disadvantages, but the spin coating sol-gel technique has more advantages compared to other existing techniques. The spin coating sol-gel technique combines physics and chemistry techniques very easily and effectively by simply adjusting the parameters of time, rotational speed and viscosity of the solution through the measurement of the heating temperature so that some research using spin coating techniques such as ZnO Thin Optical Properties [13] and

Thin Film Synthesis Sno_2 And Sno_2: Al Using Sol-Gel Spin Coating Technique On Glass Substrate And Quartz [14].

II. Materials Methods

In this study will only look up the finished sample and see the physics quality of the samples that have been made. the physical quality here is in terms of the quality of transparency, density and coating strength.

Subtract used are glass measures 20 x 20 x 5 mm. Before use of the glass substrate, the glass is washed in stages. Washing gradually aims to remove dirt and oils that stick on the glass. Leaching the first stage, the glass substrate is inserted into a measuring cup containing a mixture of water and detergent, then vibrated using stirrer for 30 minutes. After that, the glass substrate is rinsed with water until clean. Washing the second stage, the substrate is put in a glass beaker containing methanol, then vibrated with a stirrer for 30 minutes then wash with technical methanol. The glass substrate is dried in a furnace at 100 °C for 1 hour. The glass substrate is clean, then stored in plastic clips.The process of making sol-gel in this study using basic materials Tin (II) chloride dehydrate ($SnCl_2.2H_2O$ with a molar mass of 225.63 g/mol, purity 99.9%), ethanol and HF each of which serves as the solute, solvent and stabilizer (stabilizer). In this study, sol-gel materials are synthesized is divided into three (3) with a concentration ratio of (1: 99)%, (5 : 95)%, and (10: 90)%. Then mix in a measuring cup on a stirrer at a temperature of ± 80 ^oC for 1 hour, or until the solution looks homogeneous mixture.

The process begins with a layer of growth in sol-gel material drops on the substrate 0.2 ml to 5 times a trickle. Then centrifuged for 30 second with a rotation speed of 2000 rpm. Once the surface evenly coated substrate, the substrate is dried in a furnace at 100 °C for 5 minutes.

The heating process is done in three stages. First heating at a temperature of 150 °C for 1 hour, aiming to remove water and residual solvent levels in the coating gradually. A second heating at a temperature of 350 °C for 1 hour. The increase in the regulated temperature slowly from room temperature to 350 °C. This stage is regarded as the pre-heating function to remove ethanol, water, and facilitating change $SnCl_2.2H_2O$ to SnO_2. The next stage is the post-heating or heating end at 550 °C, for 1 hour. Similar to the process of pre-heating, regulated temperature rise slowly from room temperature to 550 °C. Post-heating is used to form the SnO_2 particles with a uniform crystal orientation, and eliminating pores.

III. Results And Discussion

This research made a thin film by synthesizing a thin layer of SnO_2 pure and SnO_2 with Fluorine doping with sol 1 M concentration, where SnO_2 is pure with a mixture of $SnCl_2$ compounds. H_2O with ethanol and $SnCl_2$. $2H_2O$ with doping Fluorine with variation of doping concentration of Fluorine ie 1%; 5%; and 10% using sol gel spin coating technique.

The materials used in the synthesis of SnO_2 crystal research with a thin layer of sol-gel method is Tin (II) chloride dehydrate ($SnCl_2.2H_2O$ with a molar mass of 225.63 g/mol, purity 99.9%), a total of 2.2653 gram, Ethanol C_2H_5OH, 20 ml and Hydrofluoric Acid (HF) 40%, a total 0.125 ml, 1,25 ml, and 12,5 ml.

sol gel technique consists of hydrolysis reaction and condensation reaction. Hydrolysis is a chemical reaction between precursors and water that make up other compounds, while the condensation reaction is a chemical reaction that molecules merge to form larger molecules by releasing small molecules (eg H_2O). Here is the reaction of hydrolysis and condensation in the process of sol gel :

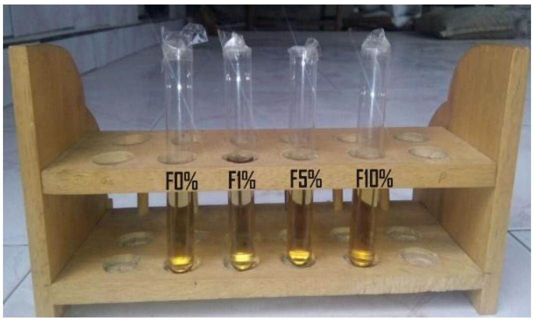
$$M(OR)_4 + xH_2O \rightarrow M(OH)_{4-x}OH + xROH(\qquad \text{Hydrolysis}$$
$$M(OH)_{4-x}OH_x + M(OR)_4 \rightarrow (OR)_{4-x}MO_xM(OR)_{4-x} + xROH \qquad \text{(Condensation)}$$

In terms of structure, metal oxides consist of M-O-R junctions, where M is metal, O is oxygen and R is an alkali group. The opposite charge of M and O will induce the formation of polarization in the M-O bonds which will make the alkoxide receive a reaction material such as water. In the presence of water, the alkoxide undergoes a substitution-addition reaction, in which the alkoxide (OR) group is released from the hydroxyl (OH) group produced by water. This process is known as hydrolysis. After the hydrolysis process, the metallic groups join together with different mechanisms, generating a metal oxide hydrate network that will eventually form a small nucleus (several nanometers).By chemical reactions occurring in the sol gel process in the preparation of pure SnO_2 and SnO_2 doping thin films F as follows:

$$SnCl_2 2H_2O + C_2H_5OH \rightarrow Sn(OH)_2 + C_2H_5Cl + H_2O \qquad \text{(pure } SnO_2\text{)}$$

$$SnCl_2 2H_2O + C_2H_5OH + HF \rightarrow SnCl_2 + C_2H_5F + HCl + H_2O(\text{F : } SnO_2)$$

Sol-gel solution for a wide range of concentrations that have been synthesized are stored in a test tube as figure 1.



(a) (b) (c) (d)

Figure 1. Results of Sol-Gel-Varying concentrations from left to right with a concentration(a) undoped, (b) F : SnO2 (1 : 99)%, (c) F : SnO2 (5 :95) % and (d) F : SnO2 (10 : 90) %.

(a)

(b)

(c)

(d)

Figure 2. Results of Sol-Gel-Varying concentrations from left to right with a concentration(a) undoped, (b) F : SnO2 (1 : 99)%, (c) F : SnO2 (5 :95)% and (d) F : SnO2 (10 : 90)%.

One thing to be determined is proving SnO_2 structure which has a tetragonal structure with crystal with unit cell parameters a = b and ≠ c, as figure 3.

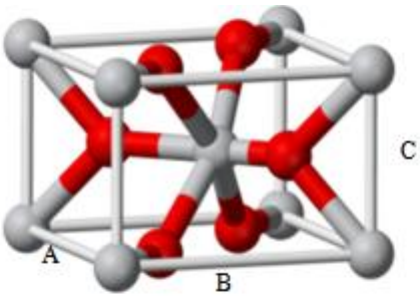


Figure 3. The model of the unit cell of the crystal rutile SnO_2

In terms of transparency the thin layer of fluorine dye fluorine SnO_2 is more transparent than the pure SnO_2 as it is seen that a thin layer of pure SnO_2 forms a rather whitish layer on the glass substrate while for Fluorine dye SnO_2 only visible spots on the substrate and transparent even in the doping layer F 10% looks a little whitish color but can still look transparent.

In terms of density we can compare between a thin layer of SnO_2 pure with SnO_2 doping Fluor, a density of a thin layer formed from a pure layer that is denser than a thin layer doped in Fluorine. This can be seen from the form of a layer where a thin layer of SnO_2 pure looks more opaque and visible dots form a less visible layer while for thin layer doping Fluorine layer more visible spots look far apart.

In terms of strength we can see from the process of thin layer damping shows that a thin layer of SnO_2 pure and SnO_2 doping F 1% seen when lifted after the damping still looks as usual while for the doping F 5% and 10% have flaked layers of the slightly opaque becomes transparent this shows that the compound F is difficult to react with other compounds, it is also proven in the process after heating for the doped SnO_2 F 5% and 10% easily removed thin layer after 4-5 times coating.

IV. Conclusion

This research is preliminary to the formation of the sample and test the physical quality of the sample. The basic ingredients used are Tin (II) chloride dehydrate ($SnCl_2.2H_2O$ with a molar mass of 225.63 g/mol, purity 99.9%), a total of 2.2653 gram, C_2H_5OH Ethanol 20 ml, and Hydrofluoric Acid (HF) 40% as much as 0.125 ml, 1.25 ml, and 12.5 ml. Sol-gel materials are synthesized is divided into three (3) with a concentration ratio of (1 : 99)% (2.2653 gram: 0,125: 20 ml), (5 : 95)% (2.2653 gram: 1.25: 20 ml) and (10 :90)% (2.2653 gram: 12.5: 20 ml). after the sol-gel material so the sample will grow on glass substrates by using sol-gel spin-coating. The physical quality of the thin film shows the quality of the Fluorine doping thinner layer better than the non-doped SnO2 thin film.

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