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Synthesis of SnO_2 Thin Layer with a Doping Fluorine by Sol-Gel Spin Coating Method

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Abstract: A thin layer of tin oxide with doping Fluorine was synthesized using the sol-gel spin coating method. The synthesis aims to determine the quality of thin layers formed based on temperature variations and the number of layers. The basic material used is $SnCl_2.2H_2O$, while the doping material used is NH_4F with variations in concentrations of dopants 0, 5, 10, 15, 20 and 25%. The substrate used is glass with a size of 10 x 10 x 3 mm. The synthesis of the thin layer includes substrate preparation, sol-gel making, thin film making, and heating process. At this stage SnO_2:F is deposited on a glass substrate with sol-gel spin coating technique at the concentration of sol 1 M with doping levels of Fluorine 0, 5, 10, 15, 20 and 25%, and treated with maturation for 24 hours. Making a thin layer using a spin coater at a speed of 2000 rpm for 3 minutes. The layer made consists of one layer, two layers, three layers and four layers. The resulting layer shows that the higher the doping percentage, the higher the transparency of the layer. In addition, the more the number of layers, the lower the transparency level.

Keywords: Thin Film, Tin Oxide, Fluorine, Sol-gel, Spin Coating

Introduction

The tin oxide (SnO_2) is one of nano-sized materials including semiconductor materials. The tin oxide is widely applied to solar cells, optoelectronic devices, transparent conductive oxide (TCO) and sensor gases. In addition, tin oxide has several advantages, which have high chemical stability, high transparency, and low resistivity, band gap width of around 3.6 eV (Schel et al, 2017). The tin oxide is also very sensitive to the presence of gas around it which makes it applied as a sensor gas (Carvalho et al, 2012).

In its application, tin oxide are known to have an energy band gap that is quite wide (~3.6 eV) so that waves are small absorbent. Because of these factors, tin oxide needs to be added to other impurities called dopants (Ikraman et al, 2017). The tin oxide is usually doped with antimony (Sb) or what is called ATO (Antimony Tin Oxide) (Hammad et al, 2011), FTO (Fluorine Tin Oxide) (Gurakar et al, 2014), AFTO (Antimony and Fluorine doped Tin Oxide) (Lin et al, 2017), and AITO (Aluminum Tin Oxide) (Doyan et al, 2018). Of the several existing doping elements, fluorine is one of the good doping elements in the thin layer tin oxide. This is because fluorine is more chemically resistant, cheaper and the availability of raw materials is easier (Adnane et al, 2005). In addition, compared to other doping elements, fluorine is more resistant to heat treatment (Sima et al, 2010).

The addition of fluorine doping has been done by several previous researchers. These researchers include: Lin et al (2017), Bakr et al (2016), Kar et al (2015), and stated that the growth of thin layers of tin oxide by doping fluorine or SnO_2:F can reduce energy band gap, increase optical transmittance and conductivity electricity, changing structure, increasing response, selectivity and stability in sensor gases.

Besides addition of doping, the factors that affect the quality of the thin layer tin oxide and SnO_2:F produced are the preparation method and the growth conditions of the layers. Methods of preparation and growth of tin oxide layers, both pure and doping additives have been carried out by several previous researchers such as sol-gel spin coating to synthesize thin layer tin oxide with Aluminum doping (Doyan et al, 2017), pyrolysis spray to synthesize thin layer tin oxide with doping Fluorine (Bakr et al, 2016), and sol-gel dip coating to synthesize tin oxide thin layers (Carvalho et al, 2012). The dip coating method is the simplest

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technique, can grow layers on various forms of substrate, but the thickness of the layer is less controlled (Doyan et al, 2017). The spray pyrolysis method has several advantages including the particle production process which is continuous, homogeneous, and uniform size distribution, but the morphology of the particles can be hollow, and the distribution of particles is uneven (Setiawan et al, 2016). Compared to the three techniques, sol-gel spin coating has more advantages, which are effective and very easy, simply by adjusting the rotational speed, time and viscosity of the solution through heating temperature measurements (Imawanti et al, 2017).

Method

This study uses the basic ingredients Tin (II) chloride dehydrate ($SnCl_2.2H_2O$) 23.702 grams with a molar mass of 225.63 grams/mol and purity of 98%. The solvent uses 20 ml ethanol (C_2H_5OH) with a molar mass of 46.07 gram/mol and purity of 98%. The material for doping is 0.555 grams of Ammonium Fluoride (NH_4F) with a molar mass of 37 grams/mol. The substrate used is glass with a size of 10 x 10 x 3 mm. Other supporting materials are aquades, detergent soap and alcohol used to clean the substrate. The thin layer synthesis includes substrate preparation, sol-gel making, thin film making, and heating process.

Preparation is done by inserting a glass substrate into a measuring cup containing a mixture of water and detergent and then vibrating with a shaker for 30 minutes. After that, rinse the glass substrate with aquades until clean, then soak it in alcohol for 30 minutes. Then drying the glass substrate using a furnace with 100^0C temperature for 1 hour.

Sol-gel preparation using variations of fluorine doping mass was 0%, 5%, 10%, 15%, 20%, and 25% respectively. The making is done by dissolving $SnCl_2.2H_2O$ and NH_4F into 20 ml ethanol with a fixed concentration of 1M, then stirrer using stirring magnetic until the solution is homogeneous. After that, the solution is left to stand for 24 hours.

Making a thin layer is done by dripping sol-gel on the glass substrate and then rotating using a spin coater at a speed of 2000 rpm for 3 minutes. After that, drying the substrate at room temperature. Next heat a thin layer using a furnace with a temperature of 150^0C for 1 hour.

Result and Discussion

Synthesis of SnO_2:F thin layer includes substrate preparation, sol-gel making, thin film making, and heating process. Substrate preparation aims to remove oil and dirt found on glass substrates.

Preparation of sol-gel made with fluorine doping variation of 0, 5, 10, 15, 20 and 25%, then in-stirrer using magnetic stirring, aiming to speed up the solution became homogeneous, then cured for 24 hours. The mass of $SnCl_2.2H_2O$ and NH_4F of each ingredient is determined by equations 1 and 2.

$$(100-n)\% = \frac{(m_t SnCl_2{\cdot}2H_2O)\ x \frac{1}{Mr\ SnCl_2{\cdot}2H_2O})}{(m_t\ NH_4F)\ x \frac{1}{Mr\ NH_4F}} \quad (1)$$

$$(n)\% = \frac{(m_t\ NH_4F)\ x \frac{1}{Mr\ NH_4F})}{(m_t SnCl_2{\cdot}2H_2O)\ x \frac{1}{Mr\ SnCl_2{\cdot}2H_2O}} \quad (2)$$

Based on the conditions 1 and 2, the mass of $SnCl_2.2H_2O$ and NH_4F was obtained as in table 1.

Table 1: Percentage of doping and number of masses

Doping Persentage (%)	**$SnCl_2.2H_2O$ (gram)**	**NH_4F (gram)**
100:0	4.515	0
95:5	4.289	0.037
90:10	4.063	0.074
85:15	3.837	0.111
80:20	3.612	0.148
75:25	3.386	0.185

Figure 1 shows that the higher the concentration of the dopant, the higher the transparency of the soles produced. This is caused by the addition of fluorine dopants can reduce the reaction of $SnCl_2$ with ethanol so that the transparency of the sol increases with increasing doping value (Ikraman et al, 2017).

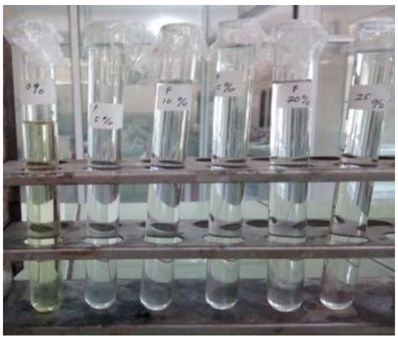

Figure 1. Sol SnO_2 for doping variation Fluorine

The next step is the process of making thin layers with doping variations and layer thickness using spin coating. Figure 2 shows a sample of

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SnO_2:F which was synthesized using the spin coating method with 150 ^{0}C annealing temperature. The higher the concentration of doping, the higher the level of transparency of the formed layer.

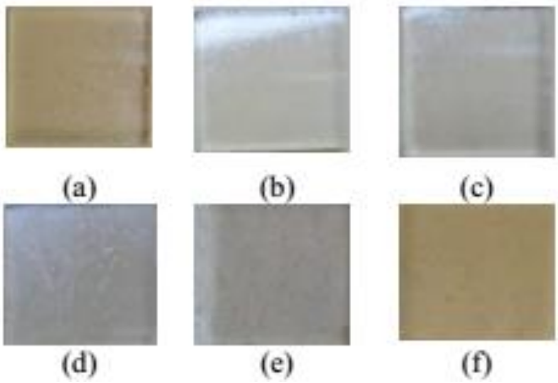

Figure 2. Thin layer with a temperature of 150 ^{o}C. Variations in SnO_2: F (a) 100: 0%, (b) 95: 5%, (c) 90: 10%, (d) 85: 15%, (e) 80: 20%, (f) 75: 25 %.

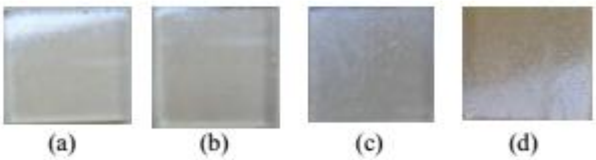

Figure 3. Thin layer SnO_2: F variations of layer (a) one layer, (b) two layers, (3) three layers, (d) four layers.

Figure 3 shows a sample of SnO_2:F synthesized using the spin coating method with various layers. The more the number of layers, the lower the transparency of the layer, so that the layer becomes more opaque. This is because the size of the grains increases as the number of layers increases (Ray et al, 2010).

Conclusion

The synthesis of a thin layer of fluorine tin oxide using spin coating sol gel technique has been successfully carried out. The level of transparency of the resulting tin oxide layer increases with increasing fluorine doping. In addition, the more the number of layers, the lower the transparency of the layer.

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