

Fabrication and Testing of SnO_2 Thin Films as Gas Sensor

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Abstract-The Aluminium and Pladinum doped thin films were deposited on glass and silicon substrates with SnO_2 coated by the sol-gel process with annealing temperature of 500°C . The structural properties of thin films are studied in detail. The X-Ray diffraction analysis reveals that film show good Crystallinity with tetragonal rutile structure and pure and Al/Pd doped SnO_2 films have the dominating peaks indicates the orientation in these planes. The resistivity of the samples is also get decreased with the increase in temperature. Due to these properties thin films are suitable for the gas sensing application.

1. INTRODUCTION

Doped SnO_2 thin films are extensively studied in the recent times due to its Crystallinity, tetragonal rutile structure and gas sensing application. Due to these properties these films becomes the major part of gas sensing mechanism. These films have the various basic applications in many technical areas such as the liquid crystal displays, gas sensor etc. As a gas sensor it find wide application in industries and general life uses. Sensors [1] are devices that convert physical or chemical quantities into electrical signals that are convenient to be detected. A gas sensor must possess at least two functions: to recognize a particular gas and to transducers the gas recognition into a measurable sensing signals. The gas recognition is carried out through surface chemical processes due to gas solid interactions. These interactions [2] may be of the form of adsorption, or chemical reactions. The transfer function of a gas sensor is depended on the sensor material [3] itself. The transfer modes employed are due to the change of thermal, mass, electrical or optical properties. However, most gas sensors give an electrical output, measuring the change of resistance or capacitance.

SnO_2 thin films are prepared by the various techniques such as RF sputtering, spray pyrolysis, chemical vapour deposition and sol-gel having its own importance. Sol gels have been used extensively as it is economical, simple and energy saving method to deposit high quality films. In this paper SnO_2 thin films are deposited on glass and silicon substrates by spin coating process. The structural and the XRD properties of thin film as gas sensor are investigated. Finally the designed gas sensor is tested in the presence of various gases.

2. EXPERIMENTAL

2.1 PREPARATION OF SOLUTIONS

Solution A-In 2.26 gm of SnCl_2 add 20cc of ethanol and stir it thoroughly. After that it is warmed on the hot plate for some time. Then do the ultrasonic procedure for 30 minutes. After that 10cc of acetic acid is added in that solution and heat that solution for 20-30 min and stir it thoroughly and now do the process of ultrasonic for 1hour. After 1 hour the solution is filtered in a container

Solution B- In 2.26 gm of SnCl_2 , add the 10cc of HCL is added and mix it thoroughly. Then warm that solution on the hot plate for 10-15 min and stir it occasionally. Now do the process of ultrasonic for 30 minutes and then add the 15cc of methanol to that solution & stir it thoroughly and add the 2cc of acetic acid to that solution to do the process of ultrasonic for 1 hour & finally filter that solution with filter paper in a container.

2.2 Doping to Solution

After the preparation of the solutions now add some impurities to that solution to increase the conductivity.

2.2.1 Solution Al

In doping process, firstly add the impurity which is aluminium chloride. In this process firstly in 2.26gm of SnCl_2 add 25mg of Al.chloride and then add 20cc of ethanol in that Solution and dissolve the solution in a container for 20-30 min stir and warm it on the hot plate. After that the process of ultrasonic is done for 30min and then add the 10cc if acetic acid in that solution and warm it for 5min and stir it thoroughly and finally do the process of ultrasonic for 1 hour and filter the given solution with filter paper in a container. Which is the desired solution of aluminium doped SnO_2

2.2.2 Solution A2

In doping process impurity which added is Pladinum chloride. In this process firstly in 2.26gm add 25mg of Pladinum Chloride and then add 20cc of ethanol in that solution and dissolve the solution in a container for 20-30 min stir and warm it on the hot plate. After that do the process of ultrasonic for 30 min and then add the 10cc of acetic acid in that solution and warm it for 5min and stir it thoroughly. Finally do the process of ultrasonic for I hour and filter the given solution with filter paper in a container. Which is the desired solution of Pladinum doped SnO₂.

2.3 DEPOSITION OF FILMS

After preparing the samples by cleaning and other processes now deposit the layers of solution on the samples of silicon and glass materials. For this we make use of the process of spin coating.

2.3.1 Layer Deposition on Silicon Samples

For cleaning piranha cleaning is used in which one part of H₂O₂ and three parts of H₂SO₄ is mixed to form a solution and in that solution the silicon samples are dipped in it for 10-15 min. then clean the wafer with by hot and cold water 3-4 times and then give the HF dip to wafers and then put them in water. After that the process of ultrasonic is used for 10-15 min and dries the sample and put them in oven at 150 degree Celsius for half hour. Then heat the wafers on thermocouple at 250 degree Celsius for 10 minutes and repeat the same process for 8 times for the better result. In this process, the spin speed of 3000rpm is used and time of coating is 30 sec.

2.3.2 Layer Deposition on Glass Samples

First of all put the solution in a biker having trylene and boil it for 10-15min and then in acetone for 10-15min. After that boil it in methanol and clean it with scrub paper. Then put the samples in nitric acid for half hour. After half hour rinse it with DI water and dry the samples and put them in oven at 150°C for 20-30min. After that we heat it on thermocouple for 250°C for 10min and finally do the spin coating process having speed of 3000 rpm and time of 30sec. And repeat the same process for 7-8 times in the presence of N₂ having temp of 500 degree Celsius for 1 hour.

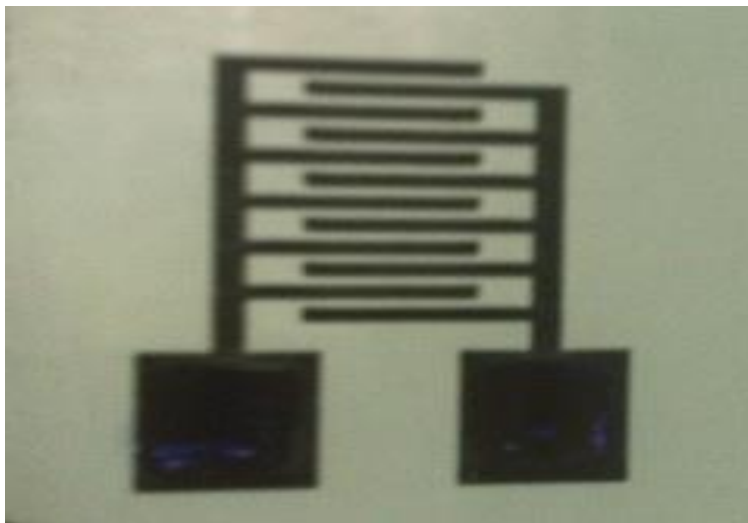


Figure 1 Designed Mask

2.4 Masking

Now after the prebaking now do the process of masking of exposure. That is the process in which alignment of the mask in desired position is done. And in this process IC mask is used which is previously designed. In this process the time which is given 1.5 minutes so that the mask is get developed to the desired position.

2.4.1. Developer

After the process of mask alignment now the process of developing is used in which the wafers are soaked in the solution of the KOH having concentration of 10% that is 10 pallets of KOH is used in 100cc of water. After this process used takes 30-40 seconds to complete it and after that the samples are rinsed in the water. After this process of post baking is done in which the temperature which is given is 100 to 120 degree Celsius and give it the time of 20-30 minutes.

When the process of post baking is done, the etching in which etchant which is 30% of the $FeCl_3$ is used and the time given is 10-12 min. and all the process is done at the room temperature. This process is called as the etching. After that in the cleaning process firstly remove the photo resist from the samples and Then dip the samples in the acetone and then also boil it in the acetone having the temperature of 60-70°C and the time which is given to it is 10 minutes after that finally rinse off the samples with the help of the DI water. And then put the samples in the oven having temp 100-120°C and the time is given to it is 1 hour.

3. RESULT AND DISCUSSION

Now after obtaining the results now we have to compare and discuss that result with the standard result and obtain the error in the experimental results.

3.1 Crystallinity

It was observed that the all the samples are highly crystalline having tetragonal rutile structure as evident from the XRD pattern in which high peaks with high intensity extended over the 2 theta scale.

Sample CH_3CH_2OH on glass sample

Table 1: Comparison of experimental result of glass sample with standard result

| Standard values Cassiterite R050502 | | Experimental values | | 2Theta %age error | d spacing %age error |
|-------------------------------------|-----------|---------------------|-----------|-------------------|----------------------|
| 2 theta degree | d spacing | 2 theta degree | d spacing | | |
| 26.63 | 3.3495 | 25.92 | 3.3127 | -2.66 | -1.08 |
| 33.93 | 2.6432 | 33.44 | 2.2326 | -1.44 | -15.5 |
| 38.00 | 2.3685 | 38.68 | 2.3584 | 1.78 | -0.42 |
| 51.83 | 1.7639 | 53.92 | 1.7423 | 4.03 | -1.22 |

Table 2: Comparison of experimental result of silicon sample with standard result

| Standard values Cassiterite R050502 | | Experimental values | | 2Theta %age error | d spacing %age error |
|-------------------------------------|-----------|---------------------|-----------|-------------------|----------------------|
| 2 theta degree | d spacing | 2 theta degree | d spacing | | |
| 26.63 | 3.3495 | 27.92 | 3.3127 | -4.84 | -1.08 |
| 33.93 | 2.6432 | 33.20 | 2.7019 | -2.15 | 2.22 |
| 38.00 | 2.3685 | 38.42 | 2.3969 | 1.10 | 1.19 |
| 51.83 | 1.7639 | 53.00 | 1.4984 | 2.25 | -15.05 |
| 66.02 | 1.4147 | 66.94 | 1.4023 | 1.39 | -0.87 |

Silicon samples in HCL solution

Table 3: Comparison of experimental result of silicon sample with standard result

| Standard values Cassiterite R050502 | | Experimental values | | 2Theta %age error | d spacing %age error |
|-------------------------------------|-----------|---------------------|-----------|-------------------|----------------------|
| 2 theta degree | d spacing | 2 theta degree | d spacing | | |
| 26.63 | 3.3495 | 26.86 | 3.2797 | 0.86 | -2.08 |
| 33.93 | 2.6432 | 33.12 | 2.6337 | -2.38 | -0.35 |
| 38.00 | 2.3685 | 37.90 | 2.1934 | -0.26 | -7.39 |
| 51.83 | 1.7639 | 51.90 | 1.6492 | 0.13 | -6.50 |
| 66.02 | 1.4147 | 64.56 | 1.3590 | -2.21 | -3.92 |

Glass Sample in HCL solution

The X-RAY diffraction analysis reveals that film show good Crystallinity with the tetragonal rutile structure and pure/Al/Pld doped films have the dominating peaks indicates the orientation in these planes due to which films are suitable for the gas sensing application. Further the resistivity of all the samples decreased with increase in temperature. The Crystallinity[6] is improved with Al doping in silicon substrate and with the Pld doping particle size is get varied. Tables given below give the comparison of XRD results of standard RRUFF (R050502) with the experimental results of both the glass and silicon samples.

Table 4: Comparison of experimental result of glass sample with standard result

| Standard values Cassiterite R050502 | | Experimental values | | 2Theta %age error | d spacing %age error |
|-------------------------------------|-----------|---------------------|-----------|-------------------|----------------------|
| 2 theta degree | d spacing | 2 theta degree | d spacing | | |
| 26.63 | 3.3495 | 27.68 | 3.2983 | 3.94 | -1.52 |
| 33.93 | 2.6432 | 33.44 | 2.6976 | -1.44 | 2.05 |
| 38.00 | 2.3685 | 39.22 | 2.2934 | 3.21 | -3.17 |
| 51.83 | 1.7639 | 52.68 | 1.6998 | 1.63 | -3.63 |
| 66.02 | 1.4147 | 67.74 | 1.3638 | 2.60 | -3.59 |

4. CONCLUSION

Pure and aluminium/Pladinum doped SnO₂ thin films were grown on the silicon and glass substrate by sol gel technique. The experimental data shows that the doped Pladinum, aluminium ions are the primary reason for the shift in the peaks of SnO₂ XRD pattern. Also the peaks at some position are stronger than the other which confirms the presence of SnO₂. Finally the specified gas sensor is designed and tested in the presence of various gases.

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