

RRST- Electronics

Construction of Versatile Advanced Micro Processor based Controller for Spray Pyrolysis Unit and Study of Characterization of Nano Crystalline Tin Oxide (SnO₂) Thin Films

V. Chaitra, K.S. Shamala, V. Uma*

Department of Electronics, Mount Carmel College, Bangalore, India

Article Info	Abstract
Article History Received : 27-06-2011 Revised : 05-09-2011 Accepted : 05-09-2011	Significant improvements in coating deposition control can be achieved with advanced micro processor based controller for spray pyrolysis coating technology that provides precise control of film thickness for the application of thin films and Photo resist to wafers. FY series Digital PID controllers are used in this system for deposition of thin as well as nano structural materials. In this paper we compare advanced system with old traditional system. This is more reliable, more economical for the laboratory and small-scale industries. Nano grain sized tin-oxide (SnO ₂) films have been prepared through microprocessor controlled spray pyrolysis unit. The polycrystalline nature of SnO ₂ film was characterized by XRD. The homogenous and continuous film was confirmed by SEM micrographs. The films exhibited transmittance, around 70%, in the visible region. The optical band gap found to be 3.6 eV. Work-hardening index was found to be 0.5, which reveals the material is hard.
*Corresponding Author Tel : +91-9341943755 Fax : +91-22286386 Email: umamcciyer@gmail.com	
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Introduction

The Spray Pyrolysis Technique has been, one of the major techniques to deposit a wide variety of materials in thin film form. The prime requisite for obtaining good quality thin film is the optimization of preparative conditions viz. substrate temperature, spray rate, concentration of the solution etc. In recent years an emphasis has been given to a variety of atomization techniques such as ultrasonic nebulisation, improved spray hydrolysis, corona spray pyrolysis, electrostatic spray pyrolysis and microprocessor based spray pyrolysis. An extensive review of thin film materials prepared during the last 10 years is given to demonstrate the versatility of the chemical Spray pyrolysis technique [1-2]. It is observed that the properties of thin films depend very much on the preparative conditions. The properties of the thin film can be easily tailored by adjusting or optimizing these conditions, which in turn are suitable for a particular application. The enhancement in deposition efficiency and improvement in quality of the thin films can be achieved with atomization techniques such as microprocessor based spray pyrolysis.

Tin dioxide, a wide band gap semiconductor with high chemical stability and excellent optical and electrical properties, has been extensively investigated for gas sensors, electro catalytic anodes and optical-conductive coatings for solar cells [4-5]. Various processing routes, both physical and chemical deposition methods, have been utilized to prepare SnO₂ thin films, including pulsed laser deposition, radiofrequency sputtering, sol-gel process, spray pyrolysis, electron beam evaporation etc., Spray pyrolysis technique is preferred among these techniques due to their low cost, simpler and more versatile than any other technique. Another

advantage of the spray pyrolysis technique is that it can be adapted easily for production of large-area films. In this work films, we have taken PART A and PART B.

Part A Explains microprocessor controlled spray unit and compared with traditional one. Part B explains Structural, optical characterization of SnO₂ prepared by spray pyrolysis.

PART A

Traditional Spray pyrolysis unit:

The fig1. Shows the diagram of the conventional spray unit. Kanthal wire was wound round the set of four ceramic tubes. A flat aluminum sheet was placed just 2-3 cm above the ceramic tubes using the clamps. The external ends of the kanthal wire were brought out of the chamber and connected to the voltage control unit panel consisted of a variac. The temperature was monitored by Fe-Cu thermocouple connected to a dummy substrate placed in close proximity to the one, which is to be sprayed. The spraying nozzle is vertically placed at a distance of 30 cm above the substrate. The chemical solution is taken in a beaker, kept outside the chamber and narrow glass tube is immersed in the solution to be sprayed. When the compressed air /gas is passed through the nozzle, fine particles of spray is formed as shown in figure.1

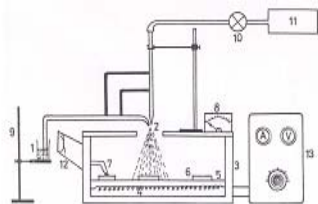


Figure 1. Spray pyrolysis unit: 1.Spray solution, 2. Nozzle for the spray, 3.Spray chamber, 4.Heater, 5 and 6- substrates, 7.Thermocouple, 8.Pyrometer, 9. Stand, 10. Pressure controller/ solution flow controller, 11.Air compressor, 12.Exhaust, 13. Variac

Microprocessor controlled Spray unit:

Present unit is a versatile FY series controller, which are microprocessor based controllers, which have been designed with high accuracy input, various output selection, useful options and good reliability at a competitive price [3]. FY600 Terminals (96mm x 48mm, DIN 1/8) consists of Power supply, control output, input, alarm, transmission, communication, remote.

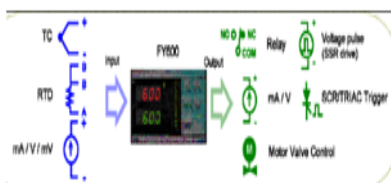
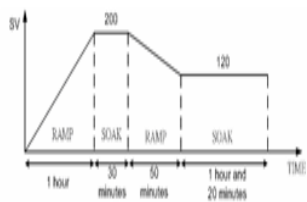


Figure2: FY series digital PID controller

It has got a unique Programmable RAMP / SOAK (Only available for PFY model) which has characteristic, that can be used for setting the temperature as well as for ramp also.



The temperature controller cum programmer circuitry essentially comprises the following subsystems viz, Precision analog lineariser for chromel-alumel thermocouple, power pack, the proportional-integral-derivative (PID) circuitry and generator.

Working: The temperature of the substrate is sensed with a temperature sensor LM335 and the output of the sensor is suitably modified with the help of a signal conditioner. The micro processor system compares the measured temperature with the desired temperature (set-point). In addition, computes the error and then maintains the desired temperature of the substrate. The system employs simple ON and OFF control technique to maintain the set point temperature. The microprocessor turns OFF the heater when the temperature exceeds the set point temperature, airflow to the spray head

are electronically controlled, providing complete control of the spray pattern shape and velocity. The coating system incorporates an advanced liquid delivery system to ensure that the coating flow is precisely metered.

Merits of advanced spray unit:

- The unit is not complicated.
- No need to set the substrate temperature, flow rate of solution and the power separately, which is time consuming.
- Tank pressure of the compressor is set automatically.
- The nozzle is made up of MS steel which has a long life and with stand the corrosion. But in the old method the nozzle is made up of glasses which are liable to break.

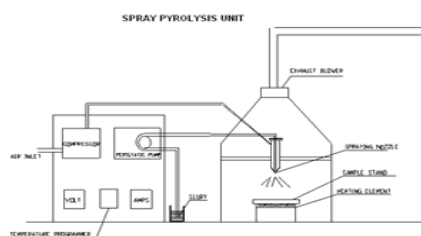


Figure 3: Microprocessor based spray pyrolysis unit

- Spray area is very large and spray inclination can be adjusted. At a time larger surface can be coated uniformly. Very much suited for industrial unit at economical cost
- Processor can be used to control temperature. Flow rate, time of spray Solution simultaneously.
- A simple heating element has been adopted in place of complicated system of ceramic tubes. The microprocessor-based system can accommodate at a time more than ten units simultaneously.
- The microprocessor based controller improves the readability of the system and makes it user friendly with on line tuning.

PART B

Experimental

Tin oxide films were deposited by a spray pyrolysis method on glass substrates. The solution was prepared from tin tetrachloride as a salt and ethanol as a solvent. The films were deposited by spraying 100ml solution through specially designed Steel nozzle on to the substrates kept at 400°C. Films prepared at 400°C exhibit a good structural, optical and mechanical properties.

Results and Discussion

Structural properties:

Structural analysis of the deposited SnO₂ film was carried out by using CuK α radiation, source having wavelength 1.5418 Å. The X-ray diffraction pattern of the film is recorded. Figure. 4 shows the XRD patterns of spray deposited undoped tin oxide thin film which is poly crystalline in nature with defined peaks that match standard interplanar spacing JCPDS card no. 05-0640 and (hkl) values are shown in figure.

The grain size of SnO₂ thin film D; can be estimated by the Debye-Scherrer formula

$D = 0.9 \lambda / \beta \cos \theta$. The average grain size of deposited film found to be 3.1 A⁰

The surface morphological study of the SnO₂ film was carried out from scanning electron microscope (SEM). It is seen from the micrograph (Figure.5) that the SnO₂ film is nanocrystalline in nature. The film prepared at 400°C substrate temperature shows the spherical type of features with higher density [6].

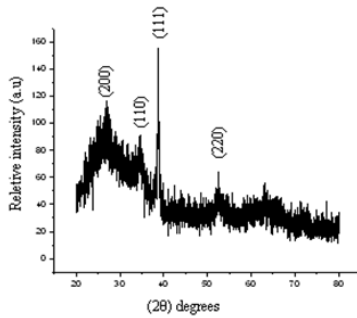


Figure 4. Shows the XRD patterns of spray deposited undoped tin oxide thin film.

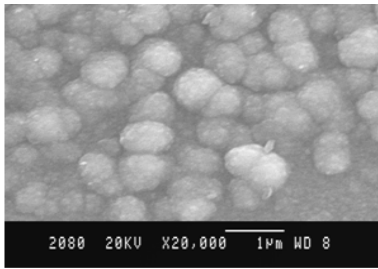


Figure 5: SEM micrographs of SnO₂

Optical properties

The optical transmittance spectra of the deposited films were recorded (figure 6).The films exhibited transmittance, around 70%, in the visible region [7, 8]. The thicknesses of the deposited films were found using maxima-minima method. The absorption coefficient was calculated from the Lambert's formula [9, 10].

$$\alpha = (2.303/t) \log (1/T)$$

Figure 7 gives the plot of $(\alpha E)^2$ versus E for the films. Optical energy gap is found by extrapolating the linear portion of the curve to $\alpha E = 0$. It was found that the optical band gap was 3.53eV and 3.6eV for S3 and S4 samples respectively.

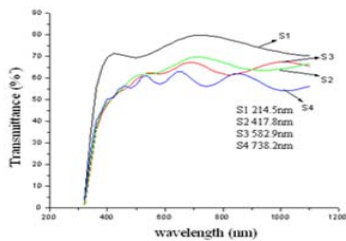


Fig.6. Transmittance spectra

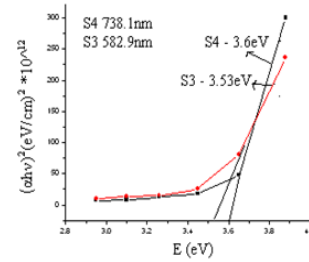


Figure 7: Plot of $(\alpha E)^2$ vs Energy

Microhardness

Micro hardness measurements were commonly carried out to determine the mechanical strength of the materials. The films were indented at different sites for the load of 50gm to 500gm for 5 second. The micro hardness is calculated using the expression [11]

$$H = 1.8544 p / d^2 \text{ Kg/mm}^2$$

The figure 8 shows the variation of micro hardness (Hmv) with load in gms for as deposited film. The non linear variation in Hardness implies the presence of imperfections and voids [12]. The imperfections such as the impurity and dislocations etc.

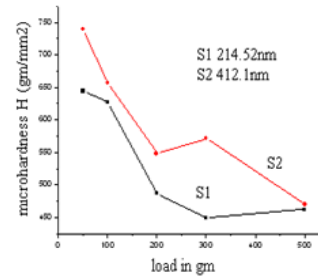


Figure 8 Variation of microhardness with respect to load

Conclusion

We can conclude, Significant improvements in coating deposition control can be achieved with advanced micro processor based controller for spray pyrolysis coating technology that provides precise control of film thickness for the application of thin films and Photo resist to wafers .we can deposit large area, uniform and defect free films in a simple manner at a lower cost for Industrial purposes. Transparent and conducting tin oxide film has been deposited by microprocessor controlled spray pyrolysis instrument.

Structural properties confirm the formation of SnO₂ film. Optical and Mechanical properties of the films studied as a function of thickness. The average grain size of the film is 3.1A⁰ at 400 °C. The observed direct band gap E_g was around 3.6 eV and average transmittance is around 70%. Work-hardening index found to be around 0.59, which reveals the material, is hard.

Acknowledgement

We wish to thank the UGC for supporting us with Major research project.

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