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"Spray pyrolysis technique use for synthesis of nanocrystalline material"

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Abstract: In this work we have report the details studies regarding the spray pyrolysis technique. Varieties of techniques have been employed to synthesis of nanocrystalline material among this technique SPT was easy to use and operate. Material waste is minimum and easy to optimized condition to obtain nanocrystalline material. Now days numerous applications of SPT thin films were performs such as gas sensing, photoconduction, optoelectronics devise as well as solar grade materials.

Keywords: Spray pyrolysis technique, Advantage and Factor Affecting on SPT,

1. Introduction

Spray Pyrolysis Equipment (Table Top) Model: VB Ceramic consultants (VBCC) is more compact and sleek model that does not take too much space in your laboratory.It can be placed in any available platform. Even though Smaller in size, it performs all the functions and operations of the standard model. The spray pyrolysis has been need of research and development and qualities control laboratories. Spray Pyrolysis System has been designed for research laboratories in thin films, especially for solar cell and gas sensor development. The deposition of thin films on different types of substrate of the size of nanometer scale is important because they play vital roles in the various fields of science and technology, including the diverse fields of electronics, optics, space science, aircraft science, defense and other industries. The system automates various fatigue and error creating processes involved in the technique when performed manually. Moreover, ergonomically designed chamber provides clean and healthy atmosphere suitable for modern lab conditions [1].

Figure 1: GPS Photograph of Spray Pyrolysis System.

Spray pyrolysis 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. The process is particularly useful for the deposition of metal oxides, nanocomposites and perovskite thin films has long been a production method for applying a transparent electrical conductor [2].

The spray system is consisted of an air compressor, a spraying nozzle and piping to connect them together. The compressor has been set to a pressure range allowing it to automatically stop if it exceeds the upper pressure value and starts again if it drops below the lower pressure value set. The air outlet of the compressor has a larger diameter than the nozzle inlet. So, we need a coupling system to join them together. The compressor outlet is connected to an air pipe and a pipe Nepal is connected to the other end of the pipe. The inlet of the nozzle is connected to a hose pipe and a female pipe nepal (inner threaded) is connected to the other end of the pipe. Then, the pipe nepal of the air pipe and female pipe nepal of the hose pipe is screwed together. The piping sequence is showed in the schematic of the system below (Figure 1).

Parameters like dispensing rate of the solution and speed of spray head movement which are difficult to control manually are controlled precisely by PC based automation. A positive displacement pump controlled by stepper motor and microprocessor is used to dispense solution as per requirement. The spray head movement is also controlled by stepper motor driven linear stages in X and Y direction. The temperature of the substrate heater plate is controlled independently through a dedicated controller.

❖ **Design Goals**

Three primary goals have set before designing this spray pyrolysis system. They are:

1) Keep the setup simple.

2) Keep the cost low.

3) Design for long term usage.

Some secondary goals are also set such as, design the system such that it is expandable, portable and can be made and maintained by using domestically available parts.

2. Standard Accessories

- ❖ **Glass Container:** Glass container holds the solution to be sprayed during the coating process. Containers of two volumes (250ml & 50ml) are available as standard accessories.
- ❖ **Spraying Chamber:** Spraying chamber having 0.25 cubic meter volume, the airtight chamber fabricated from powder coated mild steel. A heavy duty glass door, along with the magnetic lock to insure an air tight sealing at the door.
- ❖ **Spray Gun:** Spray gun is made of glass material. Having diameter of 0.5mm was positioned at a distance 30 cm above the substrate.
- ❖ **Nylon Tube:**This tube carries the solution from the glass dispenser to the spray head. Nylon is resistant to most of the chemicals which has applications in spray pyrolysis.
- ❖ **Syringe Pump:** Syringe pump is preferable to the glass dispenser for solutions which should be sprayed at lower flow rates.
- ❖ **Ultrasonic Spray Head:** The standard spray head which uses a compressed air atomization nozzle can be replaced with the ultrasonic spray head which uses an ultrasonic atomizer nozzle. It breaks the solution by vibrating its nozzle at an ultrasonic frequency (Typically 40 kHz), producing a fine spray of droplets of 50-micron average size.
- ❖ **Air Compressor**: The compressor must be powered by sockets, switched and wires rated for it specially, their current limit to avoid any short circuit, fire hazards etc. The compressor should be kept clean specially the air filter in the inlet of the compressor should be cleaned regularly. The compressor oil should be changed after three months of use. All the valves and pipes should be checked for any leakage before usage. Repair or replace if necessary.
- ❖ **Nozzle:** Keep the nozzle always clean and dust free. Never exceed the pressure rating of the nozzle. To access the needle valve of the nozzle, use the key provided in the kit. The inclination angle should be less than 45o for the nozzle to work properly.
- ❖ **Hot plate:** The hot plate must be powered by sockets, switched and wires rated for it specially, their current limit to avoid any short circuit, fire hazards etc. Don't touch the hot plate surface while on to avoid burn. Cover hot plate in aluminum foil while spraying to avoid any liquid to get into the hot plate causing short circuit.

- **Thermocouples:** 'K' type thermocouple is inserted from backside of the furnace chamber to the monitor and control the whole operation.
- **Chemicals:** Follow the safety measures associated with each chemical used during the spray deposition. Use eye protection and hand gloves while handling chemicals. Don't inhale the aerosol produced during the spraying operation. Close the door of the setup while spraying operation is running.

3. Experimental Parameters:

• Concentration: 0.05 M

• Deposition Temperature: 250-450 ^{o}C

• Pressure: 0.2-0.4 MPa

• Distance between nozzle & substrate: 20 cm.

• Flow rate: 0.7 and 1.2 mL/min.

• Deposition time: 4,7,10,15 mins.

• Nozzle inclination angle: 45^{o}

❖ Annealing Conditions:

• Annealing temperature: 500^{o}C

• Annealing time: 1 hour

❖ Specifications:

Actuator Dispensing	Stepper motor
Dispensing rate	1 - 10ml / sec.
Unit capacity	50ml & 250ml
Sprayer	
Drive speed X axis (min-max)	10 - 800mm / sec
Drive speed Y axis (min-max)	1- 12mm / sec
Sprayer traverse X - Y	200mm max.
Substrate base plate	
Dimension	150 x 150mm
Max. temperature	500° to 700 ^{0}C
Power input	230V, 50Hz
PC connectivity	Serial port (RS 232)

4. Factors affecting bonding & subsequent buildup of the coating:

- Cleanliness
- Surface area Surface topography
- Profile Temperature (thermal energy)
- Speed
- Time (reaction rates, cooling rates etc.)
- Physical & chemical properties
- Physical & chemical reactions

5. Advantages of Spray pyrolysis technique.

Apart from its simplicity, SPT has a number of advantages

- Spray pyrolysis is a simple and low-cost technique for the preparation of semiconductor thin films.
- It has capability to produce large area, high quality adherent films of uniform thickness.
- Spray pyrolysis does not require high quality targets and /or substrates nor does it require vacuum at any stage, which is a great advantage if the technique is to be scaled up for industrial applications.
- The deposition rate and the thickness of the films can be easily controlled over a wide range by changing the spray parameters.
- A major advantage of this method is operating at moderate temperature (100–500 ^{0}C) and can produce films on less robust materials.
- It offers an extremely easy way to dope films with virtually any elements in any proportion, by merely, adding it in some form to the spray solution.
- By changing composition of the spray solution during the spray process, it can be used to make layered films and films having composition gradients throughout the thickness.
- Spray pyrolysis technique has been used to prepare the thin film on a variety of substrates like glass, ceramic or metallic.

Many studies have been conducted over about three decades on SPT and the mechanism of thin film formation and influence of variables on the film formation process has been comprehensively reviewed in the literature. Due to the simplicity of the apparatus and good productivity of this technique on a large scale, it offered a most attractive way for the formation of thin films of metal oxide, metallic spinal by oxides, group I-VI, II-VI, III-VI, IV-VI, V-VI, VIIIVI, binary chalcogenides, group I-III-VI, II-II-VI, II-III-VI, II-VI-VI and V-II-VI ternary chalcogenides. Recently chemical SPT has also been successfully employed for the formation of superconducting oxide films [3-6].

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