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Research Article

Technique for the growth of stoichiometry thin films

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ABSTRACT

Thin films have a very important place in semiconductor industry. The preparation of high quality thin film of binary and ternary alloys is very challenging parameter to control stoichiometry due to appreciable difference of vapour pressure in their constituent elements. The performance of such device depends on deposition technique. Here we report a broad outlines of various techniques employed for the preparation of thin films. The study enable us to choose a very simple and convenient technique Flash Evaporation which is used to deposit a thin films of alloys without any loss of stoichiometry. In this technique a specially designed flash evaporation jig is used in which the powder material is transported by a hopper- vibrator arrangement which is attached by a variable speed electric motor .The speed of the motor is controlled by knob mounted outside the vacuum coating unit. The material from the hopper is transported to funnel for onward transmission to the pre-heated boat, which was covered by a cover. This particular design of boat is prevent the splattering and preferential loss of the volatile components. Flash Evaporation modified design reported here is convenient. Experimental set up to grow thin films of high quality with stoichiometry.

Key words: *Flash Evaporation; Physical Vapour Deposition; Stoichiometry; Thin Films ; Vacuum Coating Unit.*

INTRODUCTION

Search of new materials for use in device technology is never ending process. Discovery and study of new materials, whose properties can be tailored –made constitute the core development of solid state technology. Elemental as well as binary semiconductors have till now been extensively employed for production of various electronic and opto-electronics devices[1,2] . The various methods employed for the growth of thin films are Spray Pyrolysis[3,4] , Electroless Deposition[5,6] , Vacuum Evaporation[7,8] , Sputtering[9] , Molecular Beam Epitaxy[10] , Hot Wall Epitaxy [11], Explosion Methods[12] , Isothermic Method And Metal Organic Chemical Vapour Deposition (MOCVD)[13,14]. Most of these methods require very cumbersome expensive equipment and stringent growth conditions. Some of these deposition techniques mentioned above, however require equipment which is relatively more easily available and economically more reliable like Spray Pyrolysis, Electroless Deposition, Vacuum Evaporation and Sputtering. These methods are discussed as below:

Spray Pyrolysis:

This technique has been used for the growth of semiconductor films such as CdS , ZnS and PbS , involves spraying of appropriate chemical solutions under pressure on substrates maintained at appropriate higher temperatures . The film growth takes place due to chemical reactions on the heated substrates. Use of this technique is limited to cases where the sprayed material does not undergo chemical reaction with the substrate material. In this technique quality of the film is a strong parameter of spraying rate , size of sprayed droplets and stability of substrate temperature . In actual practice, it is usually difficult to have a strict control on these parameters and at times, this results in surface roughness and non-homogeneity in the films . While higher substrate temperature can lead to loss of more volatile components and diffusion/incorporation of atmospheric contamination unwanted reaction products in this material. This method, which appears to be potent and simple , requires stringent control of growth parameters to obtain uncontaminated films of reproducible quality. However , in practice , it is difficult to control these parameters.

Solution Growth:

The other simple technique of solution growth which has commonly been used for the growth of elemental films , has been extended for the growth of binary (PbS) and ternary (PbHgS and PbHgSe) semiconductor films. Critical control of temperature , pH value of composition of the solution is absolutely necessary to obtain proper deposition conditions for the growth of multicomponent system . It is usually not possible to deposit good quality films by this technique.

Sputtering:

The Sputtering process involves the ejection of atoms from the surface of material (the target) by bombardment with energetic particles . The ejected or sputtered atoms are condensed on a substrate to form a thin film. Though sputtering is considered one of the better suited techniques to obtain stoichiometric films, it has its own problems. One of the complexities involved in this process is the difference in sputtering yields of the constituent elements. In addition, a critical control of the deposition parameters like mass , energy and angle of incidence of bombarding atom is required to obtain good quality of films.

Thermal Evaporation:

This is oldest single source simple vacuum method of manufacturing thin films. In this method the material is evaporated from resistive heated source in the form of boat, coils or crucible. Simple vacuum evaporation of binary alloy from single source evaporation has its own limitations.

The alloy films which are obtained through an atom by atom condensation followed by subsequent recombination of constituents on the substrate surface, usually suffer a loss of stiochiometry due to the difference in vapour pressures of the elements in the alloy. This problem becomes acute with increase in number of components in the alloy system and more so in cases where high vapour pressure material involved.

Flash Evaporation:

A simple and convenient technique which can be used to deposit thin films of binary alloys , is the flash evaporation method .In this technique , a proper selection and control of evaporation boat and substrate temperature provides adequate control of stoichiometry of the films .Here , a small quantity of the muticomponent material , dropped onto the pre-heated boat , cracks instantaneously into its components which then travels towards the substrate. There is practically no time for more volatile components to be lost preferentially and give rise to the loss of stoichiometry . It is thus obvious that mixtures or alloys prepared over complete range of a particular system could be flash evaporated with out loss of stoichiometry. Characteristics of the films deposited by this technique can be controlled by proper choice of substrate temperature . Under optimum condition of growth , it may also be possible to grow epitaxal films by this method .

EXPERIMENTAL SET UP AND DISCUSSIONS:

Experimental set up used for the Flash Evaporation deposition of thin films shown in fig 1 . The system is a vacuum coating unit consists of following basic units **:**

Vacuum Chamber:

This system is provided with two vacuum chambers, one is made of glass and the other of stainless steel. We used stainless steel chamber. This chamber is fabricated from polished stainless steel having diameter of 12” and height 18”. Three circular glass windows on vacuum chamber enable visual inspection of the coating process .This chamber was placed on the base plate and it made a vacuum tight seal with base plate by means of an ‘L’ type neoprene gasket . A cooling water pipe line was cooled on the outer wall of chamber to prevent overheating especially at the chamber windows and to reduce the out gassing by circulating the water. The vacuum chamber was operated in a vacuum range of 10^{-3} to 10^{-6} mbar. The chamber enclosed the system for depositing the films by flash evaporation technique.

Pump system:

The vacuum coating system used to deposit film based on conventional oil diffusion – rotary pump combination. The vacuum chamber was evacuated by diffusion pack pump a 200litres per minutes double stage , direct driven rotary pump with an overload protection.

Rotary Drive:

The rotary drive comprised of a rotating work holder which had a diameter of 6”. It was used for deposition of material uniformly on large plane surface substrates. The work holder ring was supported by three equally spaced ball bearings one of which was spring loaded , action on the rim of the work holder . The work holder was rotated by variable speed electric motor situated on a platform inside the coating cabinet.

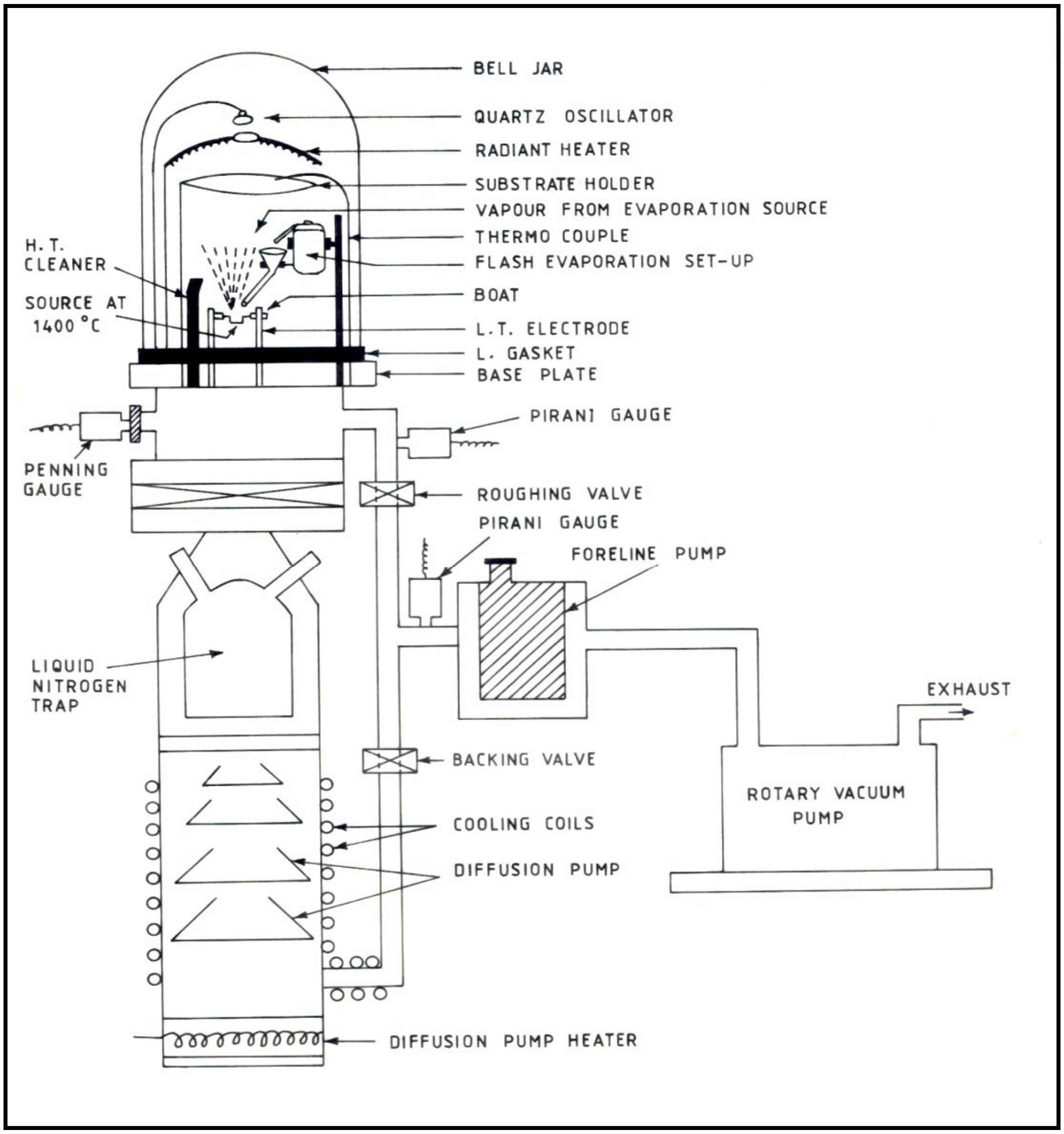

Fig 2. Λ-max for Carboplatin

Source Shutter Plate:

The plate was designed to cover the filament holder boat meant for evaporation . The movable shutter was attached to a standard source shutter shaft when a rotary drive was used . The shutter plate could be manually handled to cover or uncover the evaporation boat depending on weather the user wanted to allow the evaporant to go up from the boat or block it.

Thickness Monitor:

A quartz crystal thickness monitor was installed in an appropriate location in the vacuum chamber . Ideally , it should be located at least 250mm from the evaporation source and as close as possible to centre of substrate area . The thickness monitor allows improved manual control of the vacuum deposition process by providing a direct display of film thickness and deposition rate during deposition.

High Tension Cleaning:

High tension was also attached in the system inside the chamber. It was used to remove any absorbed vapour and gases from the chamber's internal surface. The surface was freed from absorbed gases by a high tension pressure must not fall below a specific minimum. The minimum pressure was determined by the applied voltage and distance between H.T. electrodes. In order to maintain a pressure in the chamber above this minimum while containing to pump away gases and vapours the pumping speed must be throttled and a small constant flow of air admitted to the chamber through a needle valve. Throttling was achieved by opening the baffle valve partially. Fine adjustment of pressure in the chamber during H.T. cleaning was carried out by the needle valve opening or closing.

Liquid Nitrogen Trap:

The liquid air trap was attached to the system which was optically dense and constructed out of 304 non-magnetic stainless steel plate . It was useful to obtain an ultimate and contamination free vacuum in the shortest possible time . It was designed for coolants like nitrogen and liquid or solid CO_2.

The Flash Evaporation Assembly:

A specially designed flash evaporation jig was used shown in fig 2. The powder material was transported by a hopper-vibrator which was attached by a variable speed electric motor situated on platform inside the coating unit cabinet. The speed of the motor was controlled from outside by rotating the control knob. The material from the hopper was transported to the funnel for onward transmission to the pre-heated boat .This arrangement provides a definite advantage a small quantity of the powder drops on to the preheated boat, resulting in its instantaneous (flash) evaporation. It was observed that a lot of material was lost by splattering if a simple open boat was used for flash evaporation . In addition to this the deposited films appeared to the uneven with pin holes. This could possibly be due to deposition of some very fine splattered grains of source material which got carried on to the substrate by the stream of evaporated material. It was not possible to reduce splattering by increasing the boat temperature. The problem was, however, overcome by employing a modified design of the evaporation boat. A schematic diagram of such of such a boat , made out of molybdenum sheet, is shown in fig. 2 . The boat is covered by a cover which has a 3.5mm diameter entrance aperture for the funnel and a large number (25) exit apertures of 0.75mm diameter each. The material was fed into the boat through the entrance hole. Its size was optimised to ensure that the amount of evaporant material fed into the boat through this hole is minimum . This particular design of the boat was found to almost prevent splattering and preferential loss of more volatile components.

CONCLUSIONS: Flash Evaporation is a convenient method for the preparation of stoichiometric films of materials whose constituent elements have widely different vapor pressures. In such a technique the boat temperature is maintained high enough to ensure complete instantaneous evaporation of small grains of the material fed to the preheated boat. The evaporated material condenses on substrates to provide the desired films.

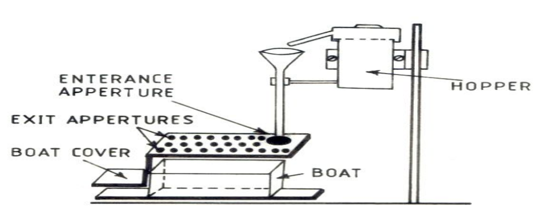

Fig.2 A specially designed flash evaporation jig

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