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Automatic Dipping And Dragging System For The Deposition of Thin Filem By SOL-Gel Method

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Abstract

Sol-gel is one of the paramount techniques to prepare micro-structured thin films. The use of automatic control arrangement has made it simpler and effective. This unit consists of a steeper motor, keyboard, and a substrate holder. The whole arrangement is executed by the microcontroller unit. The greatest advantage of this arrangement is that one needs not to be too adroit to deposit films by this method. Once the power supply has been switched on & the desired timing bas been selected by pressing the specific button in the keyboard, the substrate will dip into & withdraw from the solution automatically. Finally after rapid thermal annealing (rta) the desired film is produced. This technique is very cost effective, simple and fruitful.

Keywords: Thin film, Sol-gel, Deposition, Automatic control

Introduction

Now-a-days the Thin Film has wide applications in many sectors such as the fabrication of electronic components, especially solid-state devices and microelectronic integrated circuits, have undoubtedly found the widest and most demanding applications for thin film depositions. These films typically consist of semiconductor materials, dielectric and insulating materials, and metal or refractory metal silicide conductors. Electronic displays are used for interfacing electronic equipment with human operators. Different components and device structures are required, such as Liquid-crystal displays, Light-emitting diodes (LEDs), Electroluminescent displays, Plasma and fluorescent displays, Electro chromic displays, the fabrication of these displays requires conductive films, transparent and conductive films, luminescent or fluorescent films as well as dielectric and insulating layers, optical coatings etc. The uses of thin films are microelectronic devices, telecommunication devices, wear resistant coatings, decorative coatings, optical coatings (windows, solar cells, etc.), sensors, catalysts etc. Here Thin means less than or equal to about 1 μm Film means layer of material on a substrate So A thin film is a layer of material ranging from fractions of a nanometer (monolayer) to several micrometers in thickness. There are many deposition techniques such as spray-pyrolysis, spin-coating, anode oxidation, hydrothermal method, chemical vapor deposition (CVD), physical vapor deposition (PVD), evaporation method, sputtering method etc. Most of them have many disadvantages such as High operating temperatures, toxic Gases, generally applicable to binary and elemental films, not suitable for production of metastable products. All of them, the Sol Gel dip-coating deposition technique is most effective to deposit specially Zinc Oxide (ZnO) thin film. These films typically consist of semiconductor materials, dielectric and insulating materials, and metal or refractory metal silicate conductors. In its simplest manifestation, sol-gel dip coating consists of the withdrawal of a substrate from a fluid sol: gravitational draining and solvent evaporation, accompanied by further condensation reactions, result in the deposition of a solid film [1]. Dip Coating Systems are very useful instruments for Fabricating & Characterizing Self-assembled Structures & Multi-layered Thin Films [4].Compared with conventional thin film forming processes such as chemical vapor deposition, evaporation, or sputtering, sol-gel dip coating requires considerably less equipment and is potentially less expensive [1].

We have prepared a sol gel dip coating system especially to deposit ZnO thin film on a glass substrate. ZnO thin films have interested as transparent conductor, because the n-type ZnO thin film has a wide band gap semiconductor (Eg=3.2 eV), and high transmission in the visible range, and ZnO thin films can take place of SnO2 and ITO because of their electrical and optical properties and its excellent stability which has been mentioned widely[2][3]. ZnO ®lms used in this study were crystalline with a hexagonal wurtzite structure, the lattice constants of which were calculated to be a. 3:254 A Ê and c. 5:213 A Ê from X-ray diffraction patterns of the ZnO ®lms [5].

The sol-gel process is a wet-chemical technique widely used in the fields of materials science and ceramic engineering. Such methods are used primarily for the fabrication of materials (typically metal oxides) starting from a colloidal solution (sol) that acts as the precursor for an integrated network (or gel) of either discrete particles or network polymers. Typical precursors are metal alkoxides and metal salts (such as chlorides, nitrates and acetates), which undergo various forms of hydrolysis and polycondensation reactions [6].

The sol-gel system also called dip-coating system we have prepared which is home made, very effective, easy to control, maintenance is easy and very low cost.

Experimental Setup

The schematic diagram of sol gel dip-coating system is shown in fig-1.

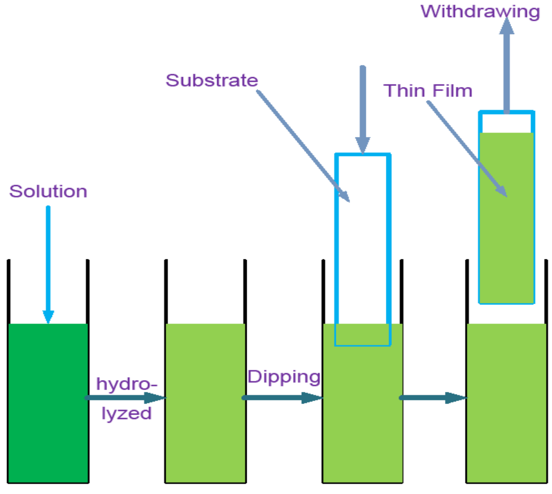

Fig.1. Dipping and withdrawing of sol-gel technique

The arrangements consists of control unit, stepper motor with dipping and withdrawing mechanism which is con-trolled by microcontroller based system, glass substrate on which film is deposited and beaker with precursor solution.

The sol-gel technique is based on hydrolysis of liquid pre-cursors and formation of colloidal Sols. The sol-gel process is particularly attractive because of the following reasons: good homogeneity, ease of composition control, low processing temperature, large area coatings, low equipment cost, and good optical properties [7]. The Sol-Gel process allows the elaboration of porous matrices with tailored nano-holes. The process starts with the preparation of a Sol, a solution composed of various required ingredients. The resultant solution was stirred at room temperature for a certain time (1/2 h). To obtain a thin film, a substrate is dipped into the Sol and withdrawn at a certain speed (like 2 mm/sec). During the withdrawal process, the Sol becomes a Gel and the micelles are entrapped in the gel network. The substrates coated with gel films were dried at 600 C before calcined at crystalline temperature. After being dried, the film was washed with ethanol to remove the surfactant molecules. The thickness of the films is adjusted by repeating the cycle from dipping to heat treatment. Finally the films are calcined with its crystalline temperature [8].

At first the substrate on which the thin film was deposited must be cleaned with ethanol in ultrasonic cleaner for 5 or 10 minutes. Then the substrate were dried by air flow throw a nozzle and then in the sunlight so that all ethanol must be dried. After preparing solution it were hydrolyzed. Then it was ready for depositing thin film on the substrate. The substrate on which thin film is deposited at first placed on the clip of dip coating system which were home made by our self so that the substrate is hanged from the clip. The beaker with precursor solution was placed on the beaker base of the device. Then the beaker was so placed that the precursor solution must be touched on the lower part of the substrate which was accompliced with level adjusting screw on the bottom of the beaker base . Then the system was ready to deposit thin film. When we press the desired key then the stepper motor start to rotate and at the same time the ribbon from the motor shaft untight so that the substrate was dipped into the precursor solution at desired rate (1cm/min). When the substrate was totally dipped the motor wait for 10 seconds so that the substrate is immersed into the solution for 10 seconds. After that the motor was rotated in the reverse direction at the same speed (1cm/min) so that the substrate was withdrawn from the precursor solution. When the withdrawn was completed, the substrate was kept from the clip of the device. The sub-strate was dried for a while at atmospheric temperature and pressure and then annealed at 4500C for 2 hours to form thin film.

In an excellent review of dip coating, Scriven states that the thickness of the deposited film is related to the position of the streamline dividing the upward and downward moving layers. A competition between as many as six forces in the film deposition region governs the film thickness and position of the stream line [1]

Dipping and Withdrawing Of Sol-Gel Technique

The block diagram of the proposed system is given in following Fig 2

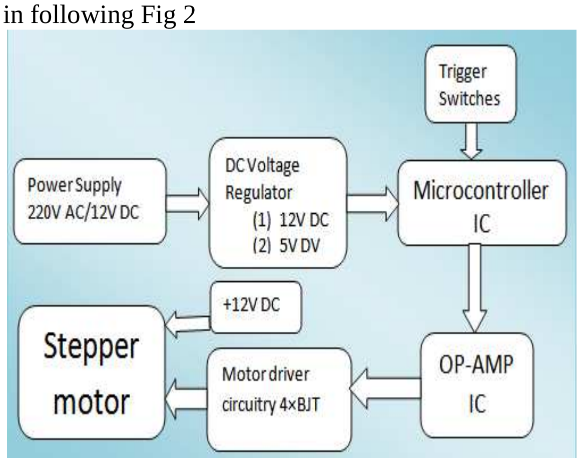

Fig.2. Dipping and withdrawing of sol-gel technique

Fig 2 shows the different stages of the operating principles of the controlling unit. At first the system will ready for taking the input from the power supply which is a transformer in this case. Then for the desired speed of the stepper motor the switch associated with every definite speed has to be pressed. After pressing the specific switch the motor will rotate primarily in the forward direction and simultaneously the substrate will dip into the solution.

But great amount of care should be taken while pressing the desired switch, because if the wrong switch is pressed the desired characteristics will contain great variation. As the dipping and withdrawing time is the same owing to the microcontroller arrangement, so the solution is spread uniformly throughout the surface and the film characteristics bear resemblance to the actual characteristics. After rotating in the reverse direction the steeper motor stops automatically. This is another inherent advantage of this arrangement because the motor is not needed to be switched off manually. So time delay can be adjusted as per our requirement between the time of end of dipping and starting of another process. If the delay is higher, then some exceptional results can be obtained which will far more different from our desired outcome. To be very honest, the delay time must not be greater than 15 seconds. Another important criterion appears in the time of heating which (heat) needs to be uniform throughout the whole process of heating. The substrate containing the solution was kept into the pot and then it was placed in the heater. Then the heater was switched on and the temperature quickly reached the 450ºc mark. We used a specially designed heater which can produce this fixed temperature. the power was not interrupted during this very period; so the substrate was continuously exposed to hating without any interruption. So, there should be a very conscious effort to stabilize the power and if the power supply is not continuous then the expected film will diverge away from the desired characteristics.

The following Fig 2 shows the block diagram of the pro-posed system. At first the conventional 230V AC supply is step down by a transformer whose voltage of two outside wires is 18 V AC. This voltage is rectified by a bridge rectifier as shown in Fig 2 which is pulse setting DC. This pulse setting DC voltage is now filtered with 1000µF capacitor. Now we have 18V DC voltage. This 18V DC is fed to the 7805 regulator to produce 5V DC regulated voltage and also fed to 7812 regulator to produce 12V DC regulated voltage. For the proposed system ATmega8 microcontroller is used to control the stepper motor. The LM 324 op-amp IC also used to switch the transistors as shown in Fig 2. In this section four Bipolar Junction Transistors (BJTs) are connected as shown in above Fig 2. Each base is connected to 10KΩ resistor. Here each collector of each Bipolar Junction Transistor (BJT) is connected to the four wire of stepper motor. The Bipolar Junction Transistors (BJTs) are operated here as switching mode.

A stepper motor is an electromechanical device which converts electrical pulses into discrete mechanical movements. The shaft or spindle of a stepper motor rotates in discrete step increments when electrical command pulses are applied to it in the proper sequence. The motors rotation has several direct relationships to these applied input pulses. The sequence of the applied pulses is directly related to the direction of motor shafts rotation. The speed of the motor shafts rotation is directly related to the frequency of the input pulses and the length of rotation is directly related to the number of input pulses applied.

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One of the most significant advantages of a stepper motor is its ability to be accurately controlled in an open loop system. The time between successive pulse is so designed that the motor shaft rotation such that the dipping and withdrawing rate into the solution is 1cm/min. In design section the target is that to rotate the stepper motor in specific direction and time so that the ribbon helps the substrate to dip and withdraw from the precursor solution to deposit thin film by this homemade sol gel system effectively and efficiently.

Design Section

The proposed homemade sol gel system is shown in Fig 3

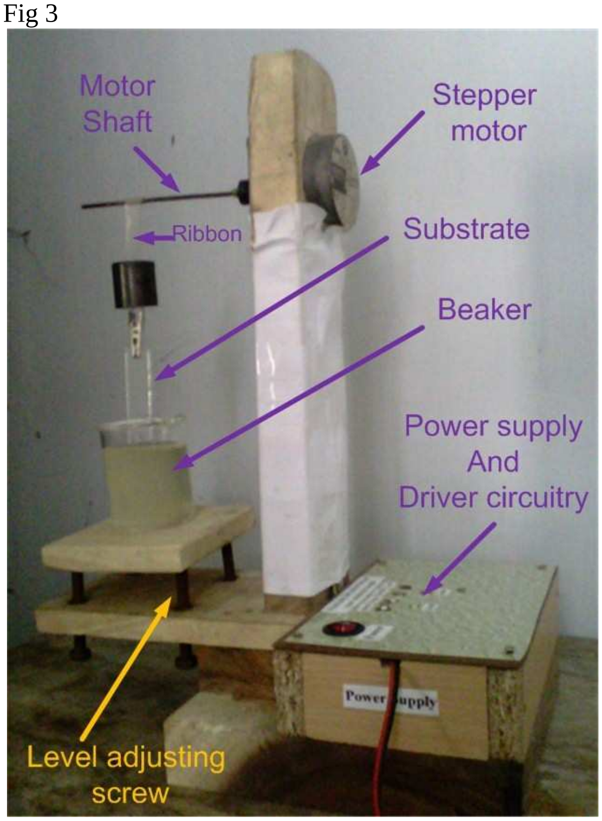

Fig. 3. Dipping and dragging of sol-gel technique

At first a wooden basement structure was designed so that the automatic control unit gets compatible with the structure. The stepper motor was placed upwards a rod of thin radius was attached to the motor shaft so that the ribbon and other accessories can be hanged. As the ribbon was too thin to be stable under any condition and the strip holding the substrate had a continuous swing in the natural wind, so a damper weight was connected with the strip to eliminate the swing and oscillation of it and thereby steadying the strip. The beaker containing the solution was placed right at the bottom & the up- down movement of the beaker was controlled by connecting screws with the wooden structure to which the beaker was being upheld. the controlling screws were fitted beneath the basement which holds the beaker. In order to deposit films of various thickness six different switches was used to make the system capable of depositing different six sizes of film. The desired time setting of each and every switch is being marked in the keyboard.

Results and Conclusion

For the desired application the substrate was heated at 450˚c for approximately two and half hours. After switching off the heater we dragged the substrate with a paper rather than the hand itself. Finally the substrate containing the de-posited film was taken into the laboratory for investigating the properties. The deposition procedure came up with great results as the Optical properties (transmittance, reflectance, absorbance and Refractive index) were really impressive as experimented in the laboratory. The transmittance was approximately 80% which is more than acceptable at this point of time. The deposited film can be effectively used in the field of material science & ceramic engineering; composition control, microstructure control, photovoltaic solar cell, protective coating etc.

Future Work

The disadvantage of our system is that before pressing any one of the six keys its strongly necessary to coil the ribbon on the motor shaft. Otherwise the substrate may not be dipped into the solution or withdrawn from the solution. In future if the circular motion of the motor shaft is converted into linear motion by using mechanical gear or pinion then the substrate dipping into the precursor solution and withdrawing from the precursor solution will be very smooth, accurate and user friendly.

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