Fabrication of Microcontroller Based Dip-Casting for Thin Films Depositions

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Citation

Abstract
An automated dip coater capable of coating thin films of less or greater than one micron from nano suspensions has been designed and fabricated using microcontroller controlled stepper motor. Various off-the-shelf components have been integrated to build a cost effective, customizable, versatile kit. The setup is used to coat thin films of photoresist material such as alumina, and poly-ethylene glycol composite on substrates to achieve uniform thickness. The automated system outperforms manual systems by allowing constant dipping speeds in the range of 10 to 100mm per second. The repeatability, reproducibility of the fabricated dip coater is excellent and the ease of use is comparable to that of commercial kits. The design considerations and selection criteria for the components are discussed in this paper. Finally, a thin film of alumina based ceramic and alumina-polymer composite thin film was made and characterized using optimal microscopy to view particles sizes and behaviours.

1. Introduction

Dip coating is a simple, industrially popular method for forming thin films. The function of the dip coater is to hold, dip and raise the substrate into and out of a suspension photoresist liquid at controlled speed so that thin films can be formed based on the viscous drag of the liquid against gravitational force. In some cases, it is advantageous to dry the samples soon after they are removed from the liquid [1]. Dip coating process can be employed in fabrication of photovoltaic solar cells, semiconductor industry and fuel cell electrolyte layers [2].

The dip coater machine consists of stepper motor, stepper motor driver circuitry and controller unit mounted on a stand. The stepper motor moves the sample from a start point to the bowl (bowl point) containing coater solution in vertical direction [3]. After dipping the sample in the coater solution, it takes the sample to the drier point which is located at a height of approximately 30 cm above the bowl point for drying the sample in the drier unit. The drier here is activated by a relay circuitry. Now the sample is again taken to the start point, where we load and unload the sample. The whole process mentioned above is fed to the controller unit PIC-Microcontroller 16F877A [4].

The sample is loaded to the dip coater with the help of sample holder which is suspended through a string. The controller unit and the stepper motor is fixed in a metal
stand which is made strong enough to withstand the vibration of the stepmotor and remain stable. Switches are given to select the mode of operation. Changes can be made in the program and burned into the IC, in order to obtain required resolution of the dip coater. For large area deposition as size poses a major limitations a dip casting is instigated and indeed surprising to observe, thinfilms as prepared in this study irrespective of the dip coating methodologies outperform their amorphous or polymer analogues owing their superior chemical and physical properties [5]. In order to obtain a microstructured thin film with desired structural and morphological properties under dip-coating process [6, 7].

Polyethylene glycol (PEG) mostly used for thin film depositions; PEG is a oligomer or polymer compound with many applications from industrial manufacturing to medicine. PEG is a less molecular mass about 20,000g/mol, having low melting solid [8]. PEG is suitable for fabrication of thin films due to its good adhesion properties, also having high depositing quality along with uniformity with precise thick and thin film. PEG films are used in pharmaceutics [14], cosmetics, and textile and leather industry for paper, plastics, rubber and printing purposes [9, 10]. PEG’s chemistry, biological amenability and excellent solubility, widely accepted the properties of PEG conjugates resides from the unique combination of physicochemical and biological properties of polymer [11, 12]. Effectively in thin film making, drug delivery and diagnosing, where it functions as a membrane through which drug is slowly released [13]. Polyethylene glycol is highly hydrophilic, its response to water vapor and its thickness can be controlled by changing the relative humidity [15,16]. Permeation properties of PEG are depends on the chemical microstructure, crystallinity, and morphology of monomer, permeant properties like, size and shape determine transport properties. The permeability depends on the solubility of polyethylene glycol, simply polymer [17].

2. Design Parameters and Tools

2.1. Mechanical Design

It consists of a metal stand to hold the stepper motor. The substrate suspended using nylon string is moved vertically from sample loading point to bowl point and in reverse i.e. from bowl point to loading point by geared spindle arrangement which is coupled with stepper motor through a shaft. The operating height is 60cm; the drier is placed in between at a height of 30 cm. The drier may be heating filament or halogen-lamp which produces heat for drying the sample and the width of the equipment is 45cm. The sample is held near the drier point for drying which is defined in the program sequence.

2.2. Electronic Design

It is used to give the required pulse to the driver unit to operate the stepper motor as defined in the program coding. Stepper motors provide a means for a precise positioning and speed control without the use of sensors. Other than the normal Microcontrollers PIC Family, the 16F877A PIC controller supports more features (discussed below), so we have chosen this PIC-controller as the main controller. The most commonly used Character based LCDs are based on Hitachi's HD44780 controller were used for our display unit. They can be interfacing with various microcontrollers. Various interfaces (8-bit/4-bit), programming, special stuff can be done with this LCD displays [18].

2.3. Supply Section

AC is applied to the primary winding of the power transformer it can either be stepped down or up depending on the value of DC needed. In our circuit the transformer of 230v/15-0-15v is used to perform the step down operation where a 230V AC appears as 15V AC across the secondary winding A bridge rectifier of four diodes (4*IN4007) are used to achieve full wave rectification and to achieve a AC to DC Conversion

2.4. Opto-Coupler and Amplifier Section

In Fig 2 we have used CNY 17-2 opto-coupler, It consists of Gallium Arsenide IR emitting diode optically coupled to a monolithic silicon photo transistor detector. A reverse biased diode is connected in parallel with the coil of the stepmotor. When the coil is demagnetizing it produce high back emf which destroys TIP122 which is in the cut off state. This can be avoided by this diode. The power transistor is used to amplify the current from milli amps to few amps (i.e. from 50mA to 2mA.)
2.5. Coding Language and Software

In this project Embedded C is used for programming the PIC (16F877A). The program consist of main program for forward and reverse movement. The sub programs which consist of delays while performing the operation. There are five sub programs delay at start point, delay at bowl point, and the delay at drier point. Display interface and relay operation at drier are called while performing the dip coating operation. Proteus version 7.6Professional simulation software was used to simulate the operation of the dip coater is showed below fig 3.

![Fig 3. Simulation part using Proteus s/w](image)

In this simulation software we can check actual working of the controller and the driver circuit by giving inputs to the simulation software. It is an easy way to test the performance of the coating technique and change the coding as we required in the simulation for future change of work. The results of the simulations help to fabricate the dip-coater which launch in way of hardware implementations.

3. Experimental Setup

![Fig 4. Dipcasting setup](image)

The Fig 4 shows the single vessel dip coater. The dip coater range is divided in two types, either single or multiple vessel systems. The single vessel dip coater is used to deposit from one solution while the multiple vessel system allows many solutions which can include cleaning or rinsing. The “single vessel” and “multi-vessel” categories both offer Small, Medium and Large systems to cater for a large range of sample sizes. You can choose from sample weight, sample size, number of samples, dipper movements and number of vessels required by changing the program.

3.1. Setup Implementation Parameters

<table>
<thead>
<tr>
<th>Sno</th>
<th>Parameters</th>
<th>Dimensions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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</tr>
<tr>
<td>2</td>
<td>Height of the system</td>
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<tr>
<td>3</td>
<td>Width</td>
<td>45cm</td>
</tr>
<tr>
<td>4</td>
<td>No of samples used</td>
<td>6 Glass Slides</td>
</tr>
<tr>
<td>5</td>
<td>Coated samples</td>
<td>Alumina-PEG</td>
</tr>
</tbody>
</table>

3.2. Glass Cleaning Procedures

Initially a glass slide (75mm long x 25mm wide), which was cleaned by using various chemicals and acids. Glass slide is rinsed in soapy water for 2 hours after again soak in concentrated hydrochloric acid (HCL), Nitric acid (HNO₃) finally acetone bath for removing the oil residues and other contaminated particles on the surface of the glass slide.
3.3. Fabrication and Characterization of Thin Films

To prepare thin films by dip coating, substrate were deposited on glass slide. Slurry was prepared by dispersing Alumina in water. Alumina (activated, neutral) was procured from Aldrich, India was a micron sized particle having wide particle size distribution. 1gm of alumina was stirred with 30 ml of water in a magnetic stirrer for 5 minutes to prepare the slurry. The slurry used for dip coating alumina-polyethylene glycol composite was made by adding 200 mg of polyethylene glycol (PEG), molecular weight ~4000 obtained from Aldrich, India. This was also stirred in a magnetic stirrer for 5 minutes and PEG being a water soluble polymer dissolved completely. This altered the viscosity of the solution and this was also used for dip coating. Dip coating was done at 30 mm/sec rate and dwell time was 5 seconds. An optical microscope, Olympus coupled with analysis software for image acquisition and analysis was used for obtaining the images.

4. Results and Discussions

Fig 5. Shows the photocopy dip coater equipment designed by us. Using the dip coater we can coat thin film of the coatersolution (egpolymer based polyethylene glycol) on a glass slide of several microns (range<1micron to few microns). The dip coater can be used to coat thin or thick film on the glass slide according to the delays defined by the user at start point, bowl point, and drier point. The thin film coated will be in the range of several microns. The resolution obtained i.e. the dipping speed in the range of approximately (30-200) mm/sec. After the deposition of chemical compositions such as alumina and polyethylene glycol kept for the drying process done by 500W halogen lamp for 10 seconds to bring film in good stability and less gluing property. The stepper motor was under ideal state for 5 to 20 seconds, which is effectively created by the delay unit.

There resolution can be changed by doing some changes such as incrementing duration for substrate bath (dipping in bowl), increasing time for drying and so on in the design of the equipment. We are using string to suspend the glass slide in the solution which will provide low resolution due to vibration of the stepper during operation using this dip coater we can obtain thin films suspension photosresist on the glass slide in the range of less than 1 micron to few microns.

Fig 6. Optical microscopy image of alumina and Alumina – PEG composite films in 50x Magnification

The thin films were analyzed for uniformity and homogeneity (lack of porosity) under the optical microscope. It is known that the as formed films have high porosity which can be reduced when sintering is done. The obtained micrographs are shown in the Fig 6. With the increase in the viscosity, thicker films are formed. Depending on the thickness and uniformity of the film required, multiple dippings may be needed.

Fig 7. Thin films of Alumina-PEG with Rhodomine dye imaged by Optical Microscope under Magnification of 20X

Rhodomine dye is added with alumina-PEG solution and kept and stirred for 30 minutes with 70°C, after then sonicating the solution for another 30 minutes for the proper dilution of alumina and PEG with concentrated dye. During deposition by extending the time for dipping the slide into the sonicated solution and characterized in optical microscope.
through objective lens (10/0.25NA) 20x magnification, in that films contain particles are get agglomerated and created a bumps size of ~20microns in range, and some of the particles are formed the grains in size of approximately 985nm and 991nm which are showed in the Fig.7. Further work is required to characterize these films in to various techniques. An automated dip coating unit with controlled dipping speed and dwell time in solution and heater zone has been fabricated along with hardware implementation.

5. Conclusion

Thin film deposition are achieved bydipping processs glass slide was simply be inserted and removed from the slurry, with help of stepper motor which was effectively controlled by Pic-microcontroller, Alumina-PEGfilm substrate get uniform deposition, with rough surface due to vibration of stepper motor, film was characterized by optical microscope with various magnification objective lenses, in that particles are forms as uncircled bubbles, some group of particles are agglomerated with the size of several microns, well diluted particles are in form of grains with various nanometered in sized, Further studies in making thin films of photocoresist and other materials are being carried out with various dilution methods by increasing stirring timings for well dilution of solvents for reducing the surface roughness, using various curing process for drying to remove the furthergluing property of the film, by varying the angle of rotation of the stepper motor possible to avoid more vibration, and characterized those samples in Near-field Scanning Optical Microscope (NSOM) and Nano Scratch Tester (NST) to get a high resolution and better accuracy compare to the present.

References

[18] www.labcenter.com