B. TECH. PROJECT REPORT

On

Design and Development of Dip Coater Unit with Simultaneous Heating of Flexible Substrate

BY

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DISCIPLINE OF MECHANICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY INDORE
November, 2016

Design and Development of Dip Coater Unit with Simultaneous Heating of Flexible Substrate

A PROJECT REPORT

Submitted in partial fulfilment of the requirements for the award of the degrees

of

BACHELOR OF TECHNOLOGY

in

MECHANICAL ENGINEERING

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November, 2016

Preface

This report on **"Design and Development of Dip Coater Unit with Simultaneous Heating of Flexible Substrate"** is prepared under the guidance of **Dr. I.A.Palani** and **Dr. Vipul Singh**.

Through this report we have tried to give a detailed design of a Dip Coater Unit with Simultaneous Heating for the purpose of coating on flexible substrates and have tried to cover every aspect of the new design, if the design is technically and economically sound and feasible.

We have tried to the best of our abilities and knowledge to explain the content in a lucid manner. We have also added 3-D models and figures to make it more illustrative.

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Acknowledgements

We wish to thank **Dr. I.A.Palani** and **Dr. Vipul Singh** for their kind support and valuable guidance.

We would also like to thank **Mr. P.Rajagopalan** for his support and guidance during our entire BTP project.

Without their help, support and valuable insight from time to time, the completion of the project could not have been possible.

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Abstract

Dip coating is one of the various methods to coat thin films on glass slabs, cylinders and many flexible substrates. The Dip coater unit along with a heater has been developed as it helps the solvent to evaporate from substrate surface. The thickness of the film is sensitive to the flow conditions and it is found that faster the substrate is withdrawn thicker is the film. This can be countered by rapid drying of volatile solution.

Polyimide is chosen as the flexible substrate because of its high thermal and chemical stability.

ZnO is chosen for coating thin films because of its abundance, biocompatibility, wide band gap energy, and high electron mobility. ZnO is prepared by using an equimolar amount of Monoethanolamine and Zinc acetate dihydrate and ethanol as solvent.

Experimental investigations have been carried out to analyse the characteristics of the thin film of ZnO coated surface of glass and polyimide substrate. The morphology, structure and optical properties of the thin film was studied using FESEM, XRD, and Photoluminescence Spectroscopy.

Keywords: Dip coater with simultaneous heating, Polyimide Substrate, ZnO Coating, FESEM, XRD, PL.

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CHAPTER 1
INTRODUCTION

1.1 What is Coating and why we need it?

A coating is a covering that is applied to the surface of an object generally referred to as substrate. In practical application one does coating to enhance the physical or chemical properties of the substrate like working temperature, corrosion and wear resistance, etc. so that it can be used in various applications. For example, in mirrors reflective coating is done and in spectacles anti-reflective coating is done. ZnO is coated because of its semiconducting behaviour, wide band gap energy etc.

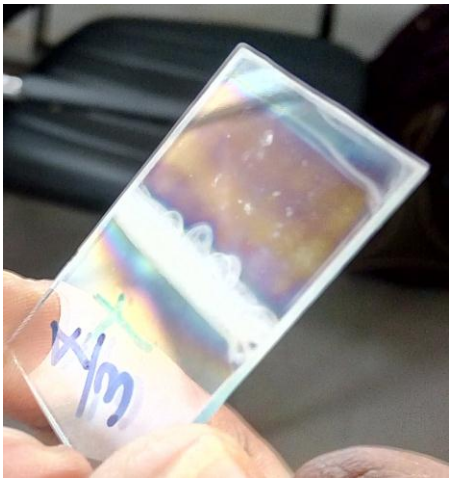


Fig 1.1 ZnO Coated Glass Slide

1.2 Types of Coating Methods

There are various techniques by which coating on a substrate can be done. Some of the techniques are:

1.2a) Spin coating

Spin coating is a procedure used to deposit uniform thin films on flat substrates. Usually a small amount of coating material is applied at the center of the substrate, which is either spinning at a low speed or not spinning at all. The substrate is then rotated at high speeds in order to spread the coating material by centrifugal force.

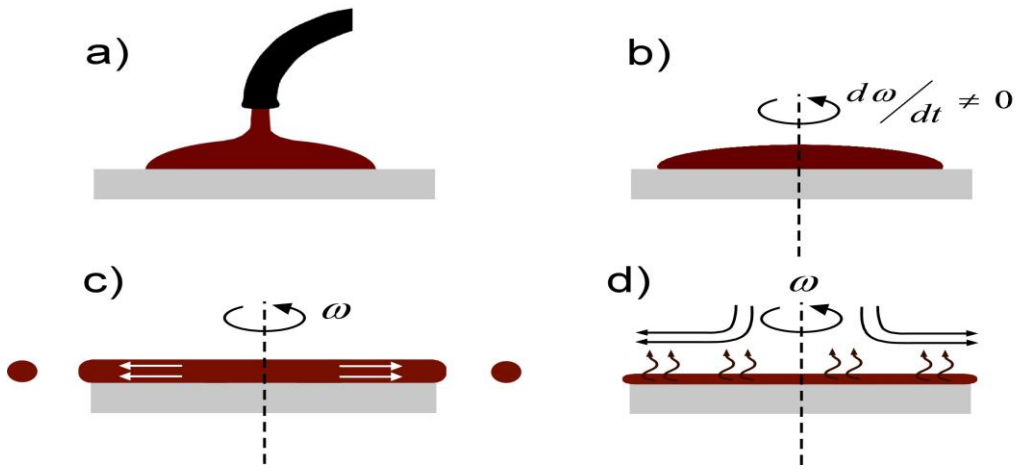


Fig 1.2: The different Stages of spin coating. a) Dispensation. b) Acceleration. c) Flow dominated. d) Evaporation dominated.

1.2b) Sputter Deposition

Sputtering is a technique used to deposit thin films of a material onto a surface (substrate). By first creating a gaseous plasma and then accelerating the ions from this plasma into some source material (target), the source material is eroded by the arriving ions via energy transfer and is ejected in the form of neutral particles - either individual atoms, clusters of atoms or molecules. As these neutral particles are ejected they will travel in a straight line unless they come into contact with something - other particles or a nearby surface. If a substrate such as a Si wafer is placed in the path of these ejected particles it will be coated by a thin film of the source material.

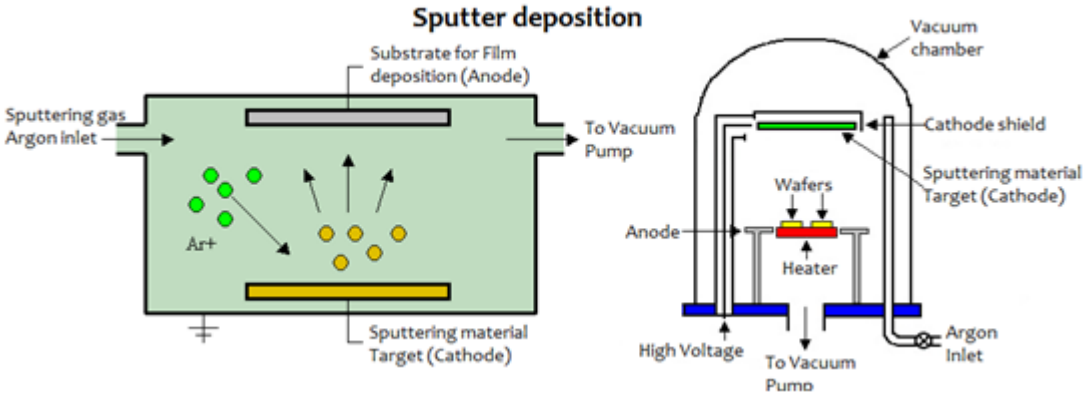


Fig 1.3 Sputter Deposition

1.2c) Flow Coating Processes

In the flow coating process the liquid coating system is more or less poured over the substrate to be coated as shown schematically in figure 1.4.

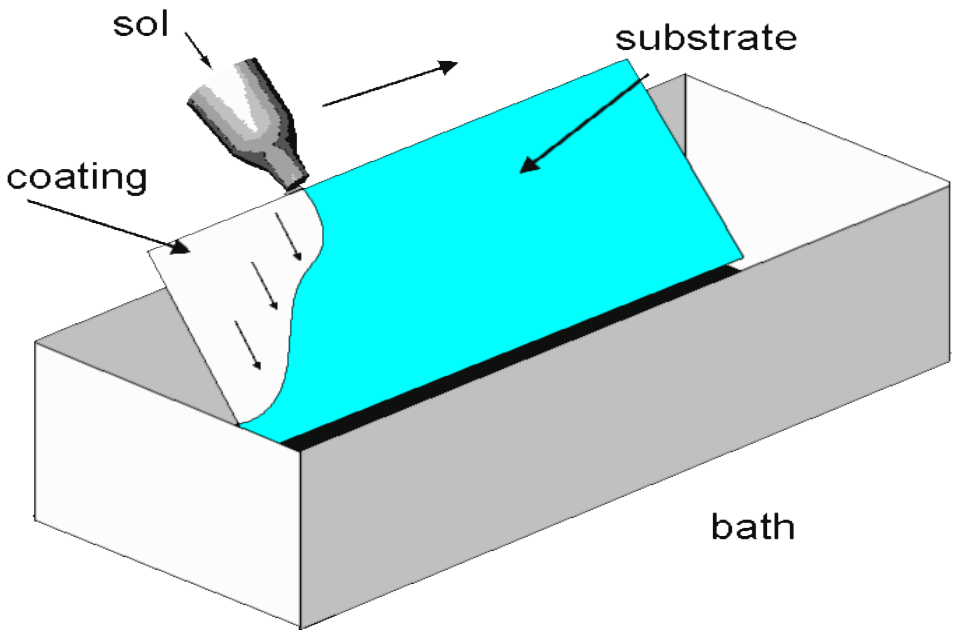


Fig 1.4 Scheme of the flow-coating process

The coating thickness depends on the angle of inclination of the substrate, the coating liquid viscosity and the solvent evaporation rate. Flow coating processes at present are used for outfitting of automotive glazing from polycarbonate with hard coating but also can be used for float glass to employ functional coatings. The advantage of the flow-coating process is that non-planar large substrates can be coated rather easily. As a variation of this process, the spinning of the substrate after coating may be helpful in order to obtain more homogenous coatings.

If no spinning process is employed, the coating thickness increases from the top to the bottom of the substrate.

1.2d) Chemical Coating

Chemical coatings should be understood as a process where a chemical reaction, e.g. the reduction of a metal is involved. The most common process is the fabrication of mirrors where the glass surface acts as a nucleating agent for the reduction of Ag^{+} to Ag^{0} in presence of reducing agent. The vast majority of all mirrors still are fabricated using this process. Another technology, which is suitable as an example for precipitating copper layers on glass, is the currently metallization process with commercially available liquids after seeding of the glass surface**.**

1.2e) Dip coating

Among the various thin film deposition methods dip coating represents the oldest commercially applied coating process. The first patent based on this process was issued to Jenaer Glaswerk Schott & Gen. In 1939 for sol-gel derived silica films. Basically the process may be separated into three important technical stages:

1. Immersion & dwell time: The substrate is immersed into the precursor solution at a constant speed followed by a certain dwell time in order to leave sufficient interaction time of the substrate with the coating solution for complete wetting.
2. Deposition & Drainage: By pulling the substrate upward at a constant speed a thin layer of precursor solution is entrained, i.e. Film deposition. Excess liquid will drain from the surface.
3. Evaporation: The solvent evaporates from the fluid, forming the as-deposited thin film, which can be promoted by heated drying. Subsequently the coating may be subjected to further heat treatment in order to burn out residual organics and induce crystallization of the functional oxides.

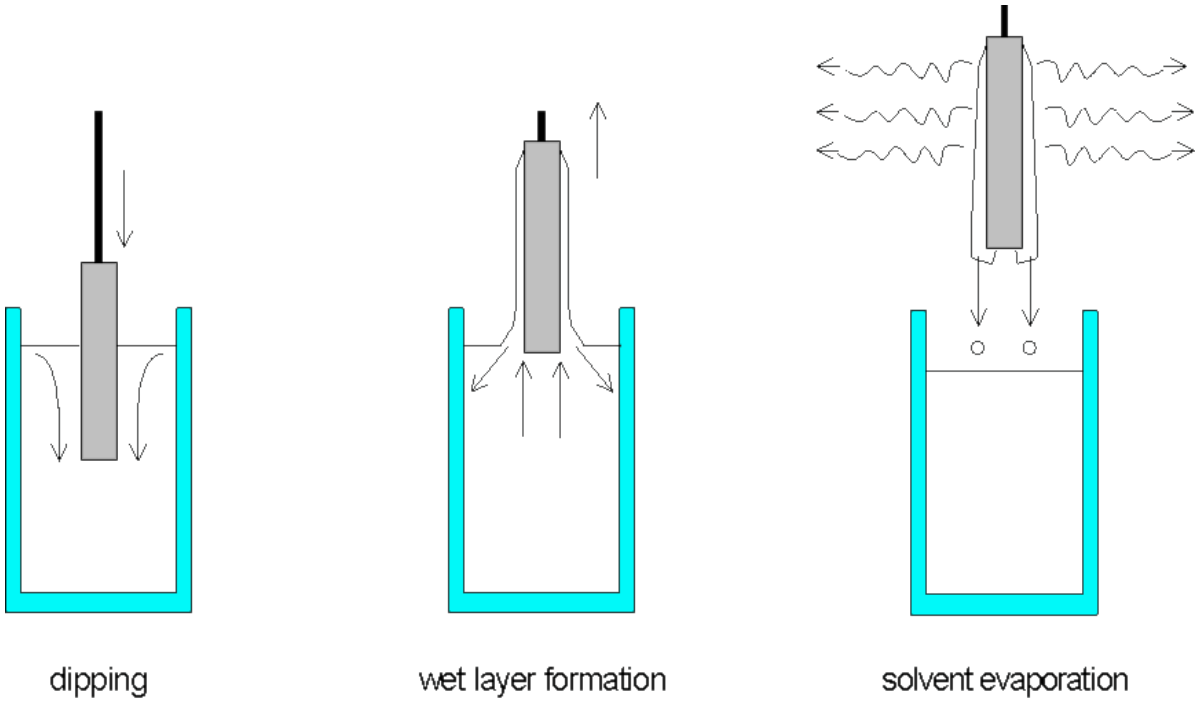


Fig 1.5 Stages of the dip coating process: **a)** dipping of the substrate into the coating solution, **b)** Wet layer formation by withdrawing the substrate **c)** Gelation of the layer by solvent evaporation

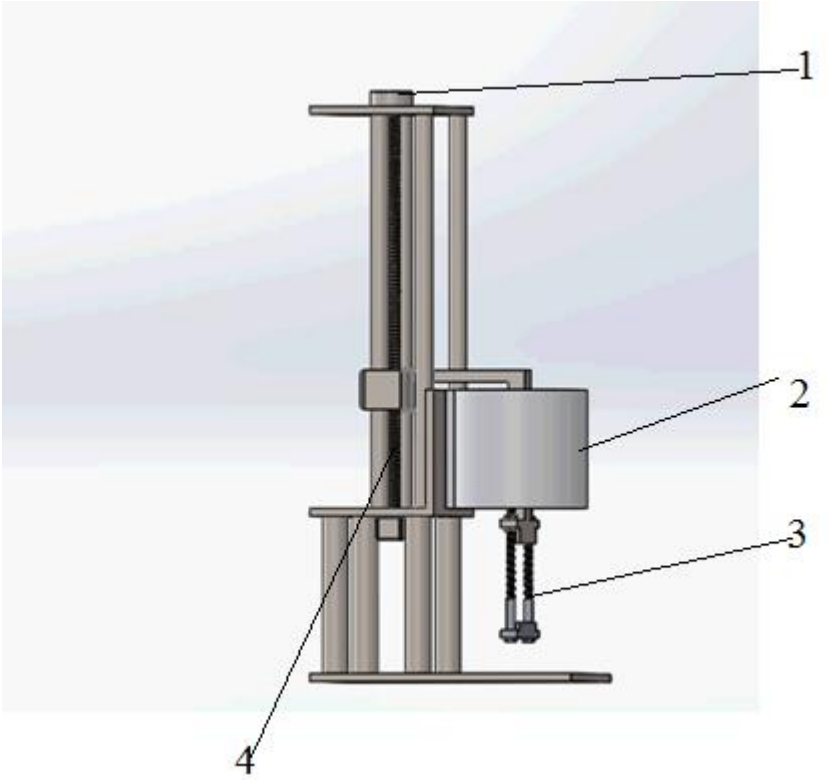
Dip-coating, while excellent for producing high-quality, uniform coatings, requires precise control and a clean environment. Coating thickness generally increases with faster withdrawal speed. The thickness is determined by the balance of forces at the stagnation point on the liquid surface. A faster withdrawal speed pulls more fluid up onto the surface of the substrate before it has time to flow back down into the solution. The thickness is primarily affected by fluid viscosity, fluid density, and surface tension. The applied coating may remain wet for several minutes until the solvent evaporates. This process can be accelerated by heated drying. In addition, the coating may be cured by a variety of means including conventional thermal, UV, or IR techniques depending on the coating solution formulation. Once a layer is cured, another layer may be applied on top of it with another dip-coating / curing process. In this way, a multi-layer stack is constructed.

CHAPTER 2

DESCRIPTION OF THE SETUP

2.1 Overall Structure

The design of the structure of the dip-coater unit was done in SolidWorks. The figure given below depicts the Solidworks model of the dip-coater unit.



1 Stepper Motor

2 Heater

3 Substrate Holder

4 Lead Screw

Fig 2.1 Solidworks Model

2.2 Material Used

Aluminium was used as the material to design the overall structure because of its low density and therefore low weight, high strength, superior malleability, easy machinability and excellent corrosion resistance. The required slots on the plate was done by CNC milling machine.

2.3 Translation Motion

Gears are more expensive to manufacture and their lubrication requirements may impose a higher operating cost per hour. Gear pairs always have some backlash and hence they require lubrication. The backlash of a gear train equals the sum of the backlash of each pair of gears, so in long trains backlash can become a problem. In both gears and belts the way to alter speed and force is through the size of two interacting wheels.

The disadvantages of pulleys, in contrast to machines that use rigid objects to transfer force, are slipping and stretching. A rope will permanently stretch under tension, which may affect the future performance of a device. If a line becomes slack, then the operation of a machine may change entirely. Also, ropes will slip and stick along pulley wheels just like belts.

So lead screw was used to get the required translatory motion because of the following advantages:

- It has large load carrying capability.
- It is compact.
- It has a large mechanical advantage.
- It gives Precise and accurate linear motion.
- It is smooth, quiet, and it requires low maintenance.
- Most lead screws are self-locking.

Specification of Lead Screw:
Length = 500mm, Pitch= 2mm, Lead= 8mm

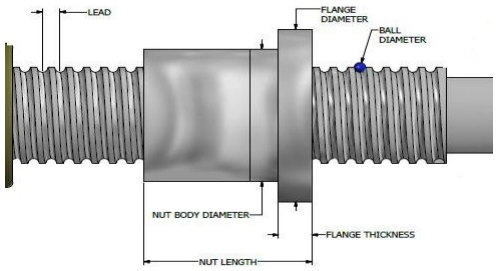


Fig 2.2 Lead Screw

2.4 Calculations

For trapezoidal lead screw raising torque is given by $T_R = F\frac{d_m}{2}\frac{(\mu\pi d_m \sec\phi + L)}{(\pi d_m - \mu L \sec\phi)}$

Assuming μ= 0.23 (For Steel Screw & Bronze Nut) , d_m=8mm, m=1kg , Ø=15°(Lead Semi angle) , L=2mm

Lowering Torque = $T_L = F\frac{d_m}{2}\frac{(\mu\pi d_m \sec\phi - L)}{(\pi d_m + \mu L \sec\phi)}$ = 0.093 kgcm

$\mu\pi d_m \sec\phi > L$ (Self Locking)

Calculations for Micro stepping

Let N= RPM of motor

v = linear velocity of substrate

d = distance travelled by substrate

p = pitch of the lead screw

No. of steps per revolution =6400

Velocity is given by (v) = Np

Time for 1 revolution = 60/N

Time for 1 step = 60/ (N*6400)

Hence, 1 step time = (60*p)/ (6400*v)

In one rev of motor substrate linear movement = 2mm

In on rev no of step = 6400;

Resolution = 2/6400 = .3125 μm

2.5 Motor to rotate the Lead Screw

The major requirement for the motor was low vibration, precision and easy control for proper coating thickness on the film. So stepper motor was selected because of its following attributes:

1. Precise positioning and repeatability of movement since good stepper motors have an accuracy of 3 – 5% of a step and this error is non-cumulative from one step to the next.
2. Excellent response to starting/stopping/reversing.
3. Very reliable since there are no contact brushes in the motor. Therefore the life of the motor is simply dependant on the life of the bearing.
4. The motors response to digital input pulses provides open-loop control, making the motor simpler and less costly to control. This type of control eliminates the need for expensive sensing and feedback devices such as optical encoders.
5. It is possible to achieve very low speed synchronous rotation with a load that is directly coupled to the shaft.
6. A wide range of rotational speeds can be realized as the speed is proportional to the frequency of the input pulses.

NEMA 17 with a maximum attainable torque of 4.2 kg cm and 5A micro stepping drive was used as a motor. It is designed for smooth and quiet operation without compromising on torque and control at higher speeds. It has short-circuit protection for the motor outputs, over-voltage and under voltage protection and will survive accidental motor disconnects while powered-up.
It achieves microstepping using a synchronous PWM output drive and high precision current feedback and this is absolutely silent when the motor is stopped or turning slowly. It virtually eliminates stopped-motor heating regardless of power supply voltage.

Its key features are:

- Smooth and quiet operation at all speeds and extremely low motor heating.
- Industrial grade performance for 2-Phase Bipolar.
- Input supply voltage from 12VDc to 40VDC.
- Selectable peak coil current from 0.5A to 2A.
- Selectable half-current during motor standstill to further reduce motor heating.
- Selectable micro-steps up to 6400 steps per rotation for a 1.8deg stepper motor.

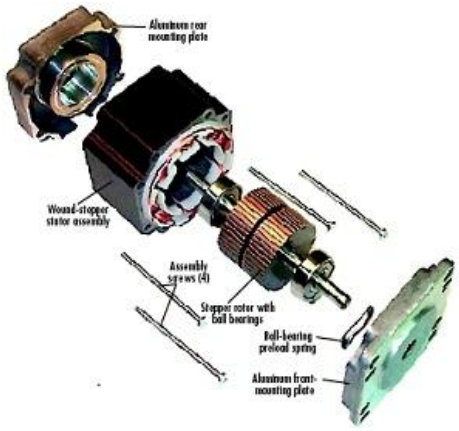


Fig 2.3 Stepper Motor

- Higher motor torque and higher speeds achievable due to advanced loop control algorithm.
- Pulse, Direction and Enable inputs with opto-isolated interface.
- Short-circuit protection for the motor outputs, over-voltage and undervoltage protection.

2.6 Drying Unit

2.6 a) Heater

The heater was a hollow cylinder with inner diameter 80mm and outer diameter 130mm and a Height of 140mm.It was insulated using Ceramic insulator and covered by steal sheet. Nichrome Wire coil was used as a heating element

Most heating elements use nichrome 80/20 (80% nickel, 20% chromium) wire, ribbon, or strip. Nichrome 80/20 is an ideal material, because it has relatively high resistance and forms an

adherent layer of chromium oxide when it is heated for the first time. Material beneath this layer will not oxidize, preventing the wire from breaking or burning out.

2.6b) Thermocouple

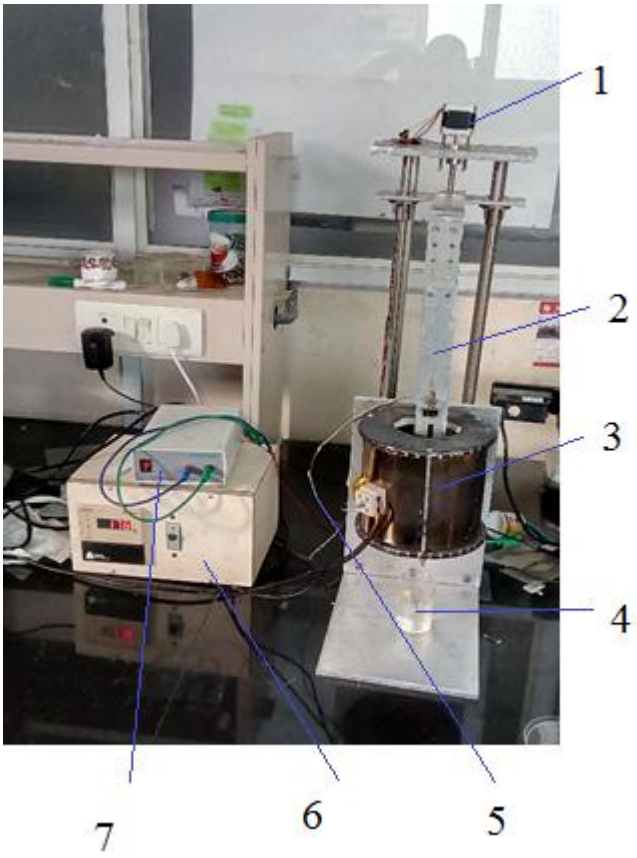
J-type Thermocouple was used to measure the temperature in the heater since they are inexpensive, interchangeable, are supplied with standard connectors, and can measure a wide range of temperatures. In contrast to most other methods of temperature measurement, thermocouples are self-powered and require no external form of excitation. Type J (iron –constantan) has a more restricted range (−40 °C to +750 °C) than type K, but higher sensitivity of about 50 µV/°C.Hence J-type was preferred.

2.6c) Temperature Controller

As the name implies, a temperature controller is an instrument used to control temperatures, mainly without extensive operator involvement. This temperature control system accepts a temperature sensor such as a thermocouple as input and compare the actual temperature to the desired control temperature, or setpoint. It will then provide an output to heater.

Fig 2.4 Temperature Controller

2.7 Setup of Dip Coating Unit



1 Stepper Motor
2 Substrate Holder
3 Heater
4 ZnO Solution
5 Thermocouple
6 Temperature Controller
7 Power Supply

Fig 2.5 Setup of Dip Coating Unit

2.8 Feature of Dip Coating Unit

1. Speed can vary from 1mm/sec to 100 mm/sec.
2. Resolution .3125µm.
3. Stroke Length - 250mm.
4. Inbuilt Temperature control system.
5. Temperature variation from 25^0C to 300^0C.
6. Speed as well as distance travelled can be changed at any point.

CHAPTER 3

EXPERIMENTAL SECTION

3.1 Materials and Sample Preparation:

3.1a) ZnO Solution

Zinc acetate dihydrate ($Zn(CH3COO)_2.2H_2O$) was used as a starting material, 2-methoxyethanol and monoethanolamine (MEA) were used as a solvent and stabilizer respectively for the solution preparation. Zinc acetate dihydrate was first dissolved in 2-methoxyethanol and then MEA were added, at room temperature. The molar ratio of MEA to zinc acetate dihydrate was maintained at 1.0 and the concentration of zinc acetate dihydrate was 0.375 M. The solution were stirred at 80°C for 2 hours, thus obtained clear solution was then allowed to cool to room temperature.

Fig 3.1 ZnO Solution

3.1b) Polyimide Film

Metal-coated polymers are used in various technologies for a wide range of applications. Thin film metallized polymers are used extensively in radio and electronics, cryogenics engineering, computer technology, solar-energy converters, etc. With the increase of the use of metallized polymers in the microelectronic industry, an understanding of the mechanism responsible for adhesion between metals and polymers has become of increasing importance. Among the polymers suitable for microelectronic applications, polyimide has received a great attention due to their thermal and chemical stability, low dielectric constant, high electrical resistivity, and relative ease of processing into coating and films. For all these reasons, polyimides have been widely used in microelectronics as dielectric spacing layers, protective coatings and substrates for metal thin films, replacing traditional inorganic insulators such as SiO_2 in many applications. In these and other applications good adhesion between polyimide and a metal substrate is required.

Fig 3.2 Polyimide Film

3.1c) Surface treatment of Polyimide film

The adhesion of metals directly to the polyimides is usually poor due to the inertness of polyimide surfaces. Failure to activate a polyimide surface will normally cause the subsequent coatings to be poorly adhered and easily cracked, blistered, or otherwise removed. Various surface treatments and modification methods have been used to enhance the metal to polyimide adhesion.These include the uses of ion beam, photografting, plasma, and sputtering. Most of these methods require high vacuum equipment and the productivity is low; thus they are not economically feasible. These methods may also introduce foreign materials and undesirable modified layers into the interfaces, resulting in possible reliability failure.

Interests in wet-process surface modifications of polyimides have increased due to simplicity and low cost. Polyimide films are resistant to most solvents and chemicals, but they react with oxidizing or reducing agents. However, if the concentrations of the chemical reagents, reaction temperatures, and reaction time are well controlled, the reactions can be confined to the surface.

Polyimide surfaces are modified in order to improve the adhesion between the polyimide and the metal substrate. Polyimide prepared from pyromellitic dianhydride and oxydianiline was treated with KOH aqueous solution and HCl to yield polyamic acids at the surface (Figure 3.3)

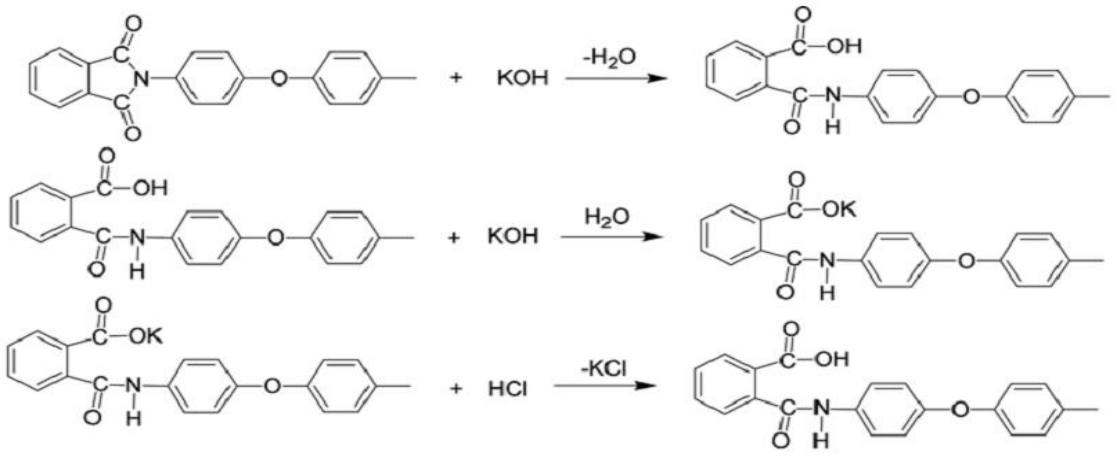


Fig 3.3 Imide ring opening in polyimide film by KOH treatment at room temperature.

3.1d) Surface treatment of Glass Slide

To improve hydrophilicity and adhesion of glass slide. Glass slide was treated with NH_3, H_2O_2 and DI water in ratio of 1:1:5 respectively.

3.2 Preparation of ZnO thin film

Different samples of glass slides and Polyimide films were coated with ZnO using Dip Coating Unit at withdrawal speeds of 3 mm/sec and 5 mm/sec. Before coating with ZnO, the substrate was preheated at 210^0C for 10 min in a heating chamber. It was kept inside the solution for 5 min such that optimal adhesion took place between the substrate and the solution. The substrate was then withdrawn from the solution at above given speeds. After that it was kept motionless inside the heating chamber for post heating at 210^0C for 10 min so that evaporation could take place.

On completion of the process of coating the substrate, some samples were annealed at 350^0C for an hour inside the oven.

3.3 Characterisation:

The morphological, structural and optical properties of the ZnO coating layers were characterized using Field-Emission Scanning Electron Microscopy (FESEM), X-ray Diffraction (XRD) and Photoluminescence Spectroscopy (PL). SEM images were captured with a ZEISS Ultra+ 55 field-emission microscope. Prior to the collection of SEM images, ZnO coated substrate was polished using gold to make the samples conductive. XRD spectra were recorded according to the Bragg−Brentano configuration with a Bruker D8 Advance diffractometer using the $CuK\alpha_1$ radiation. The XRD patterns were collected in the range of 20° to 80° owing to the very thin film thickness.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Photoluminescence

Photoluminescence is a term used to designate various effects like fluorescence, phosphorescence, Raman scattering etc. Photo means photon and luminescence means emission of light. So, photoluminescence means emission of light from a form of matter by absorption of photons.

If a matter is exposed to electromagnetic radiation then the radiation can be absorbed, transmitted, reflected or it may undergo photoluminescence.

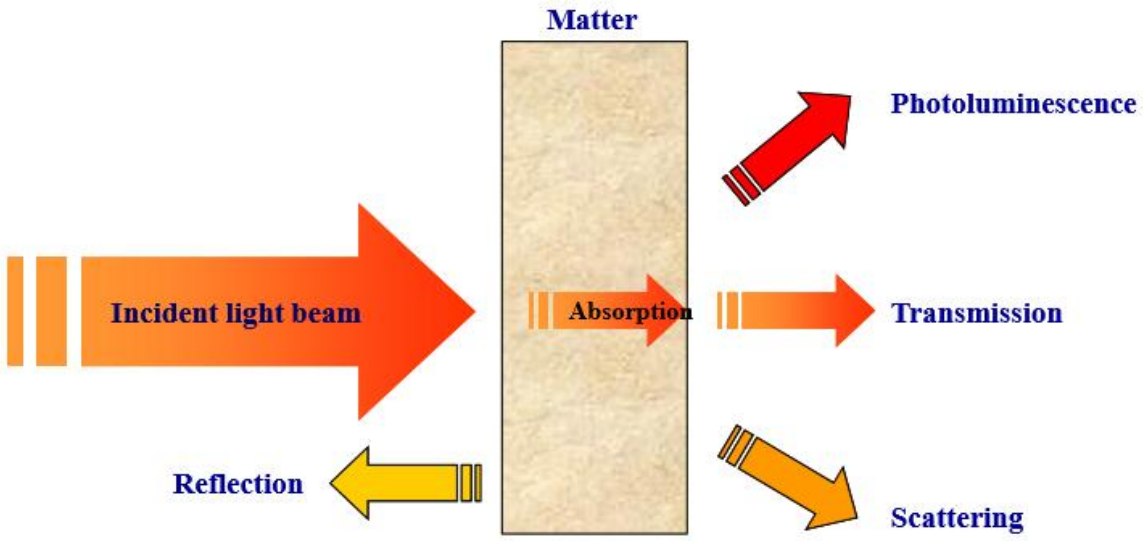


Fig 4.1 Interaction of radiation and matter

4.1a) Working of Photoluminescence (PL)

When the LASER hit the surface of the matter the electrons in the valence band absorbs energy and get excited to the conduction band and after sometimes when it de excites and comes back to the valence band it emits light corresponding to the wavelength.

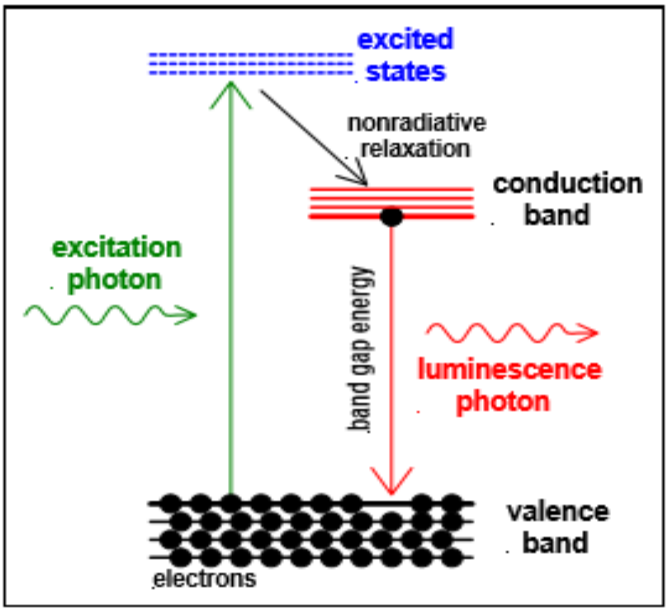
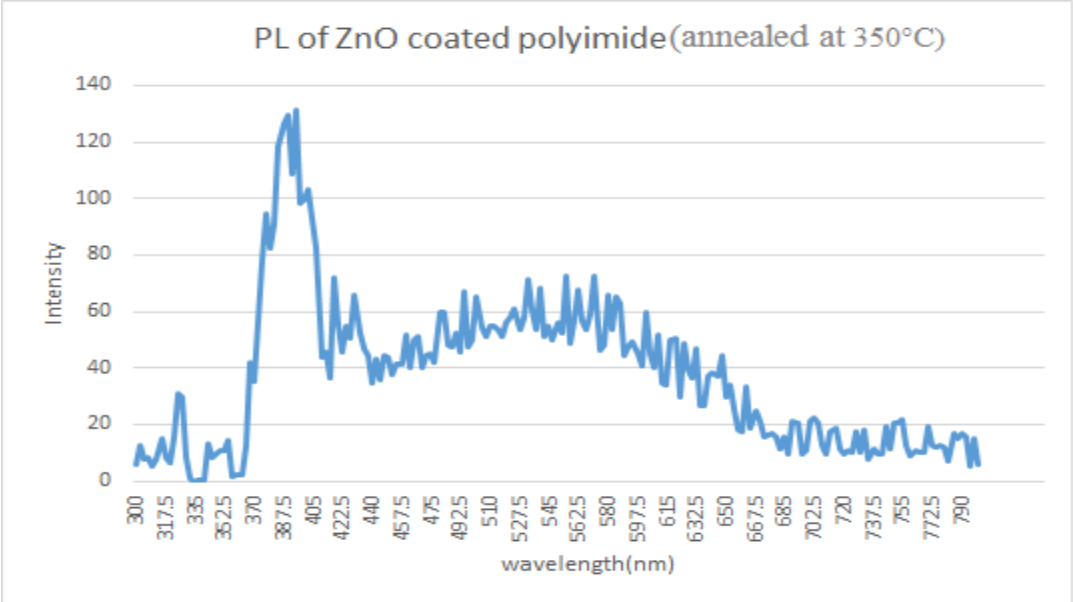


Fig 4.2 Luminescence effect

4.1b) Experimental Result



Coating of ZnO on Polyimide film at a withdrawl speed of 3mm/sec

Fig 4.3 Photoluminescence of ZnO coated polyimide film annealed at 350^0C

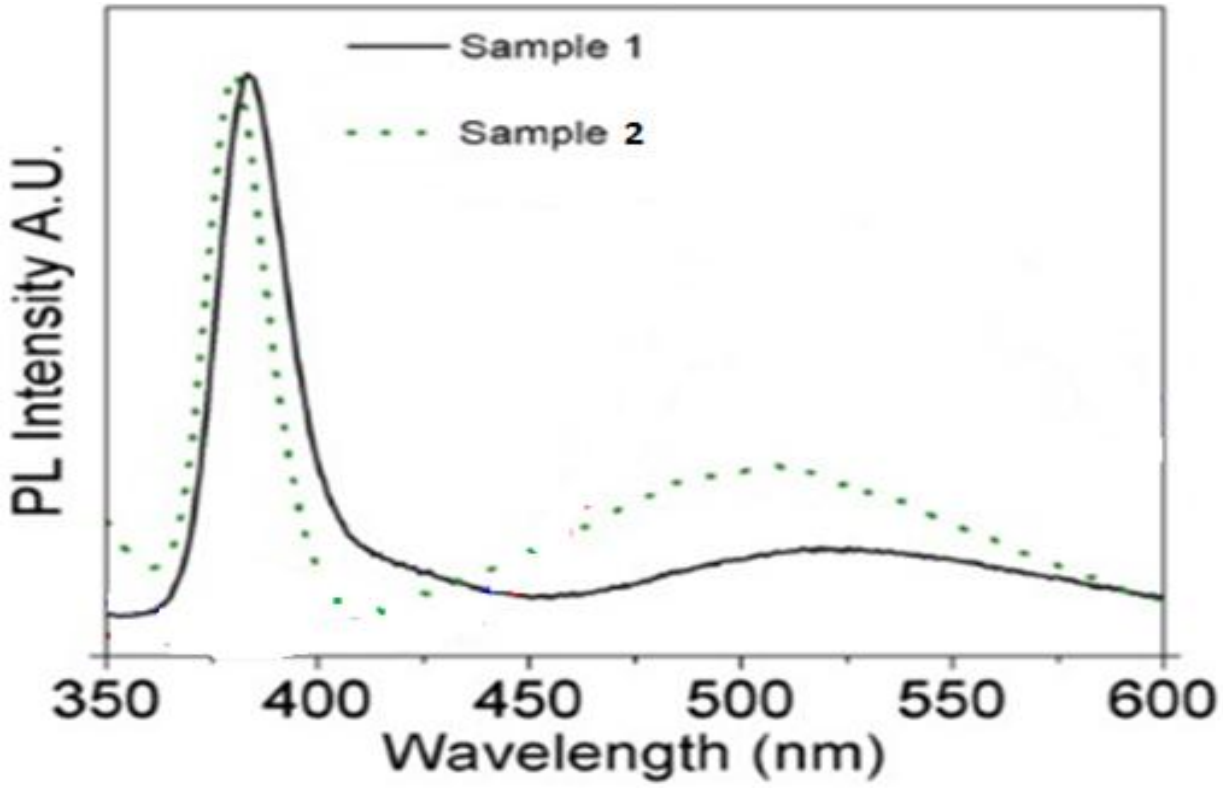


Fig 4.4 Photoluminescence of ZnO coated polyimide substrate

Ben E. Urban,[1] Jie Lin,[1] [1]Department of Physics, University of North Texas, 1155 Union Circle, Denton, TX 76203, USA

As we can see in figure 4.3, peak intensity which we are getting corresponds to the wavelength around 390 nm. Many other researchers have done Photoluminescence spectroscopy (PL) on coated substrate have got the peak around 375 nm to 400 nm. Figure 4.4 is given by **Ben E. Urban & Jie Lin** have got the peak corresponding to the wavelength around 380 nm.

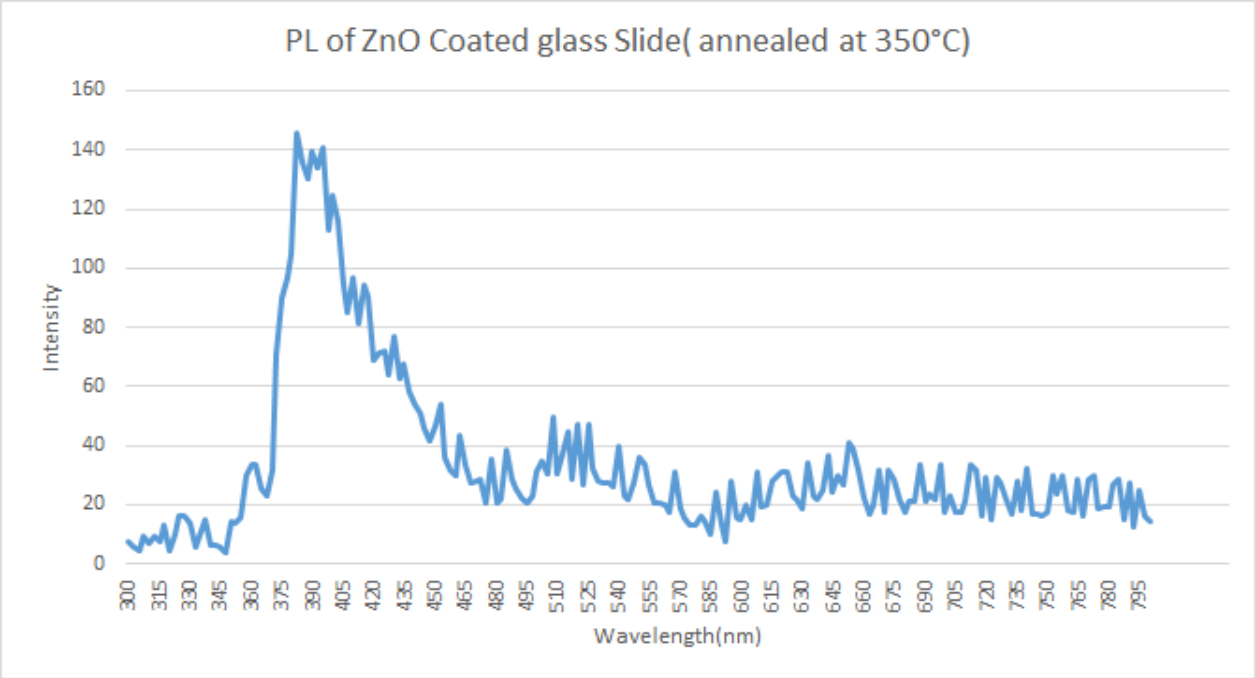


Fig 4.5 Photoluminescence of ZnO coated Glass slide(annealed at 350^0C)

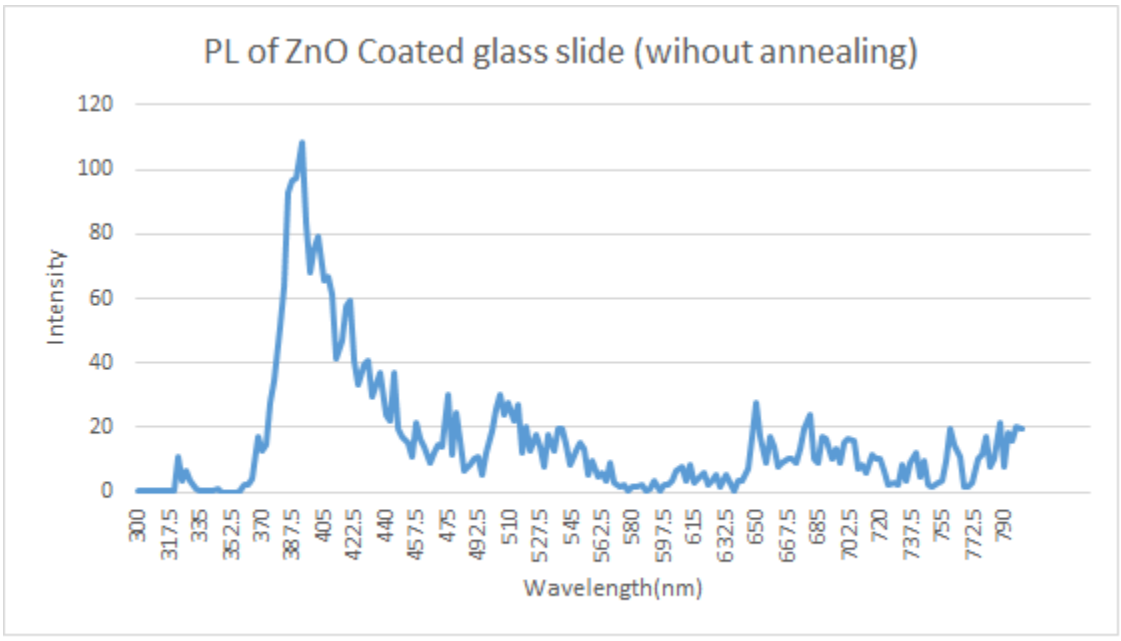


Fig 4.6 Photoluminescence of ZnO coated Glass Slide (without annealing)

4.2 X-ray Diffraction (XRD):-

X-ray diffraction is an analytical technique primarily used for phase identification of crystalline material and can provide information on unit cell dimension. Analysis of diffraction pattern allows the identification of phases within the given system.

4.2a) Fundamental Principle of XRD

X-ray diffraction is based on a constructive interference of monochromatic X-ray and crystalline structure. These X-rays are generated by cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and directed to the crystalline sample. The interaction of the incident ray with sample (a diffracted ray) generates a constructive interference when condition satisfies the Bragg's law.

BRAGG'S LAW :- $n\lambda = 2d \sin\theta$

This law relates the wavelength of the electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted rays are then detected, processed and counted. By scanning the samples to a range of 2θ angle, all possible diffraction directions of the Lattice should be attained due to the random orientation of the sample. Conversion of d spacing allows to understand the crystalline structure of the sample.

4.2b) Experimental Results

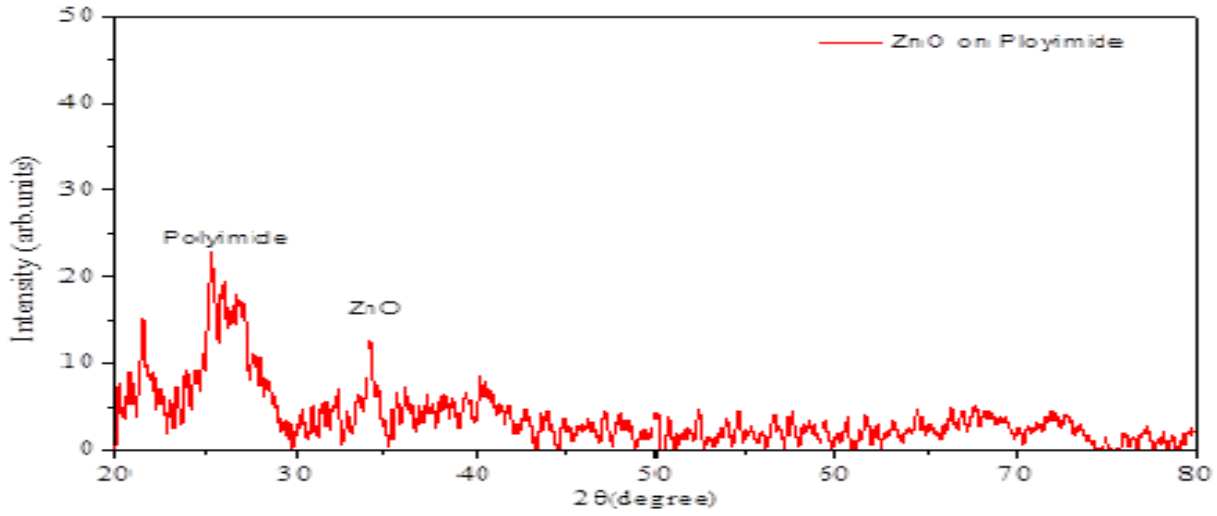


Fig 4.7 XRD of ZnO coated Polyimide

In figure 4.7 we are getting peak around 34.80 angle which corresponds to (002) plane

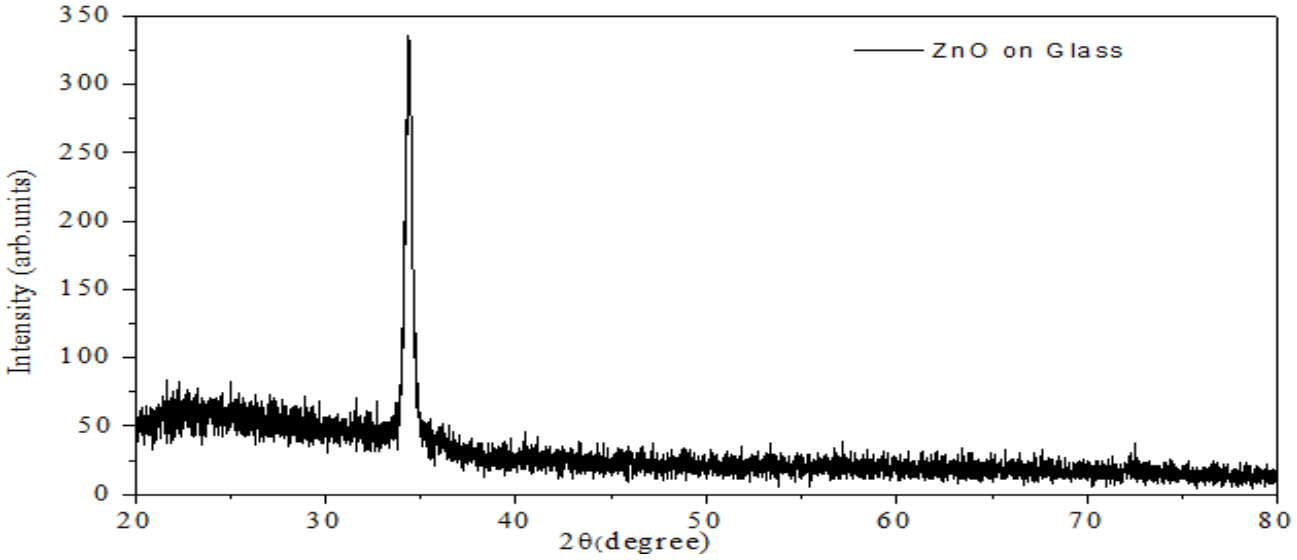


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Fig 4.8 XRD of ZnO coated Glass Slide

From figure 4.8 we can infer that coating of ZnO is highly oriented in one plane that is (002).

4.3 Field Emission Scanning Electron Microscopy (FESEM)

It was invented by Erwin Wilhelm Muller. Field emission scanning electron microscope (FESEM) is a microscope that works with electrons instead of light. The object is scanned by electrons according to a zigzag pattern. FESEM provides morphological and elemental information at magnification of 10x to 30,000x. It is an analytical technique used in material science to investigate the morphology and various electronic properties.

1. **Electron optical column**
2. **Specimen chamber**
3. **EDS detector** [Electron Dispersive Spectroscopy]
4. **Monitors**
5. **BSD** (Back scatter detector)
6. **Personal Computer**
7. **ON/STANDBY/OFF button**
8. **Plinth**
9. **WDX (not in existing FESEM)** [Wavelength Dispersive X-ray analysis]

Fig 4.9 Overview of FESEM System

4.3a) Working Principle of FESEM

In FESEM, electrons are liberated from a field emission source and accelerated in a high electric field gradient. Within the high vacuum column these so called primary electrons are focused and deflected by electronic lenses to produce a narrow scan beam that bombards the object. As a result secondary electrons are emitted from each spot of the object. The angle and velocity of these secondary electrons relates to the surface structure of the object. The detector catches the secondary electron and produces an electronic signal. The signal is amplified and transformed to a video scan image that can be seen in a monitor or to a digital image that can be further processed.

In order to be observed in SEM the objects are first made conductive for current. this can be done by coating very thin layer (1.5-3.0 mm) of gold or gold palladium.

4.3b) Experimental Results

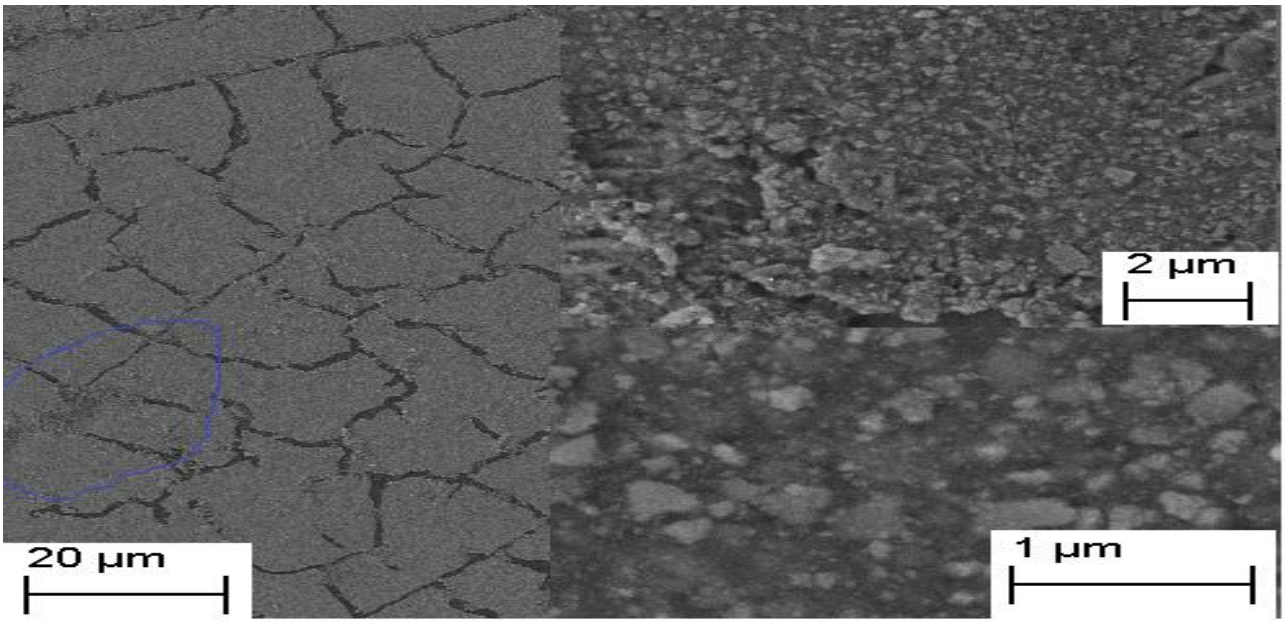

Fig 4.10 FESEM image of ZnO Coated Polyimide Substrate

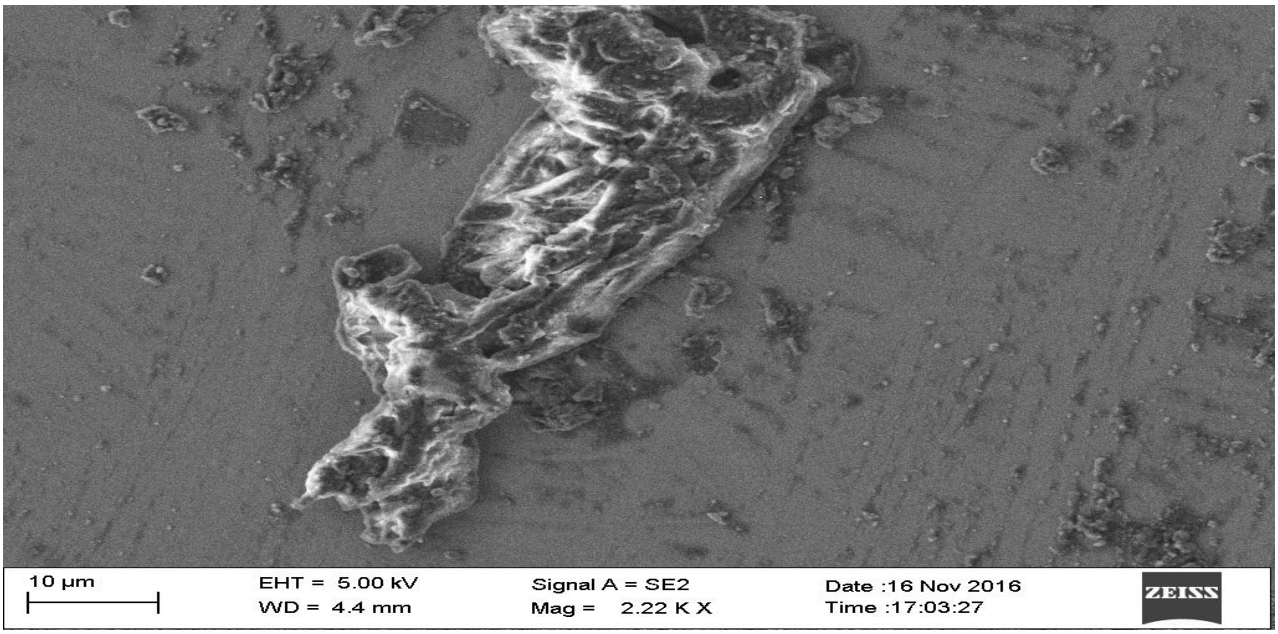

Fig 4.11 FESEM image of ZnO Coated Glass Slide

CHAPTER 5

CONCLUSIONS

This Project is aimed towards the design and development of a Dip Coating unit. There are various kinds of Dip coating unit commercially available which have their own applications and shortcomings. So we designed a Dip coating unit according that was most suitable to our requirements. In general, for preheating and post heating the substrate is taken out of the holder and heated in a oven which is a time consuming and inefficient process. So we designed it using a heating chamber such that preheating and post-heating can be done simultaneously. In our design we prepared a holder in which flexible thin film can be tightly fixed. In our actual setup Lead screw was used to provide linear motion to the substrate holder because of its precision and accuracy. Stepper motor was used to provide rotary motion to the lead screw because of its precise step control and accuracy. The stepper motor was controlled using arduino microcontroller and micro stepping driver. Using the above setup we were successfully able to achieve the required speed and temperature needed for thin-film coating on the substrate.

We prepared ZnO solution and coated it on polyimide and glass substrates. After that we studied the morphological, structural and optical properties of ZnO coating layers by field-emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and Photoluminescence Spectroscopy (PL).After comparing the results of the PL and XRD tests conducted on the coated substrates; we can say that we were able to coat ZnO on polyimide and glass substrate successfully.

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