B. TECH. PROJECT REPORT

On

THIN FILM BASED ELECTRONIC DEVICES PREPARED VIA PRINTING TECHNIQUE

BY

Vishwajit Sangle(140002030)
Kalash Agrawal(140002015)

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THIN FILM BASED ELECTRONIC DEVICES PREPARED VIA PRINTING TECHNIQUE

A PROJECT REPORT

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

of
BACHELOR OF TECHNOLOGY
in

ELECTRICAL ENGINEERING

Submitted by:
Vishwajit Sangle(140002030)
Kalash Agrawal(140002015)

Guided by:
Dr. Vipul Singh
Dr. I.A. Palani

INDIAN INSTITUTE OF TECHNOLOGY INDORE
DECEMBER 2017

CANDIDATE'S DECLARATION

We hereby declare that the project entitled **"Thin film based electronic devices prepared via printing technique"** submitted in partial fulfillment for the award of the degree of Bachelor of Technology in Electrical Engineering completed under the supervision of **Dr. Vipul Singh, Associate Professor, Electrical Engineering Department and Dr. I.A. Palani, Mechanical Engineering Department,** IIT Indore is an authentic work.

Further, we declare that we have not submitted this work for the award of any other degree elsewhere.

Signature and name of the students with date

CERTIFICATE by BTP Guides

It is certified that the above statement made by the students is correct to the best of my/our knowledge.

Signature of BTP Guides with dates and their designation

Preface

This report on **Thin film based electronic devices prepared via printing technique** is prepared under the guidance of **Dr. Vipul Singh and Dr. I.A. Palani.**

Through this report we have tried to give a detailed design of a device fabrication technique for large scale production of flexible devices and try 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 models and figures to make it more illustrative.

Vishwajit Sangle
Kalash Agrawal
B. Tech. IV Year
Discipline of Electrical Engineering
IIT Indore

Acknowledgements

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

It is their help and support, due to which we were able to complete the design and technical report.

Without their support this project would not have been possible**..**

Vishwajit Sangle
Kalash Agrawal
B.Tech. IV Year
Discipline of Electrical Engineering
IIT Indore

Abstract

Since the discovery of the polyacetylene conductivity in 1977 by Shirakawa, MacDiarmid and Heeger, Organic Electronics has been growing and establishing for a new generation of electronic devices. On one hand, the unique properties of polymeric semiconductors and conductors, such as flexibility and transparency, allow the fabrication of low-cost devices over large area: the most common are the Organic Light Emitting Diodes (OLEDs) and the organic photovoltaic cells. On the other hand, much effort has been made to investigate new technologies and processes for the realization of high-performance organic transistors and sensors. Among them, Inkjet Printing is a promising technique which exploits all the advantages of organic materials, such as low-cost and solution processability, and allows the large-scale automated fabrication of large area devices.

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CHAPTER 1

Introduction

The term *polymer* refers to a large Carbon-based molecule composed of many repeated units, the *monomers*. Polymers were historically known to have an insulating behavior: their conductivity decreases at low temperatures, same as semiconductors, while for metallic materials it increases, leading to superconductivity.

Together with electrical conductivity, also optical properties of organic materials were investigated giving rise to the organic optoelectronics. Actually, the first working polymer-based devices were Organic Light Emitting Diodes (OLEDs), realized both by means of small molecules and long-chain polymers . The comprehension on matter-radiation interactions and the progress on the fabrication process allowed the realization of devices with even higher efficiency since the early 1990s: the first devices were based on the Schottky-type diode , but donor-acceptor based systems were soon revealed to be a more promising route to generating separate photo induced charges . Finally, composites of a donor and an acceptor forming a bulk hetero junction system prevailed and still nowadays these are the most employed and advanced solution. The importance and the potentiality of Organic Electronics have been revealed to all the world, more than to the scientific community, when in 2000 Shirakawa, McDiarmid and Heeger were awarded with the Nobel Prize for Chemistry, since their work on electrical conductivity in polymers is considered as the fundamental step to the development of branch of knowledge .

Physical and chemical basics of organic electronics

Conduction in Carbon-based molecules

Molecular Orbital theory (MO), based on the orbital of the whole molecule, *i. e.* the wave-function of all the electrons which move around all the nuclei. The best approximation which leads to the molecular orbital is the *Linear Combination of Atomic Orbitals (LCAO)* theory, which defines the molecular wave-function[a](~*r*) as a linear combination of the atomic wave functions $\tilde{A}$(~*r*).On one hand, if they sum up, a molecular orbital is obtained with its maximum in the internuclear axis: this is called bonding orbital σ. Also, since the location probability of the negative-charged electrons is maximum between the positive-charged nuclei, the potential energy of the molecule is lower than that of the two dissociated atoms and the σ bond is stable. On the other hand, if the atomic orbitals are subtracted, *i. e.* they are combined with opposite signs, an antibonding orbital σ^* is obtained. In this case the molecular orbital has a node surface in the internuclear axis, leading to a higher potential energy and to a less stable system with rispect to the two isolated atoms. From two single atomic orbitals two molecular orbitals, *σ* and σ*, are formed. Extending this reasoning, the number of molecular orbitals is equal to the number of atomic orbitals linearly combined to produce them. Moreover, for the MO-LCAO only two half-filled orbitals can overlap and form covalent bonds: therefore in theory a Carbon atom in its base configuration 1*s*22*s*22*p*2 should be able to create only two bonds, but this does not justify the fact that Carbon can have up to four bonds, as in methane (CH4).

Organic molecules are composed of many atoms, and especially Carbon, connected by covalent chemical bonds. When a polymer is considered, the energetic structure is obtained from the bonding and anti-bonding orbitals of the single atoms. Although polymer chains can be large and consist of many repeated units, their energy levels cannot be as numerous as those of the crystal Silicon: in this case, it is not possible to consider them as continuous bands, but only as discrete levels. Anyways, these are divided in bonding levels and anti-bonding levels. The highest bonding level, referred as the *Highest Occupied Molecular Orbital* (HOMO), and the lowest anti-bonding level, referred as the *Lowest Unoccupied Molecular Orbital* (LUMO), are separated by an energetic gap. A direct comparison with the upper limit of the valence band *EV* , the lower limit of the conduction band *EC* , and the band gap *EG* can be done.

When two atoms are bound, the energy separation between bonding and anti-bonding levels is much higher for *σ*-*σ** orbitals than for -σ* orbitals. When many atoms build up a polymer, the energy gap, *i. e.* the energy difference between LUMO and HOMO, is much higher when single *σ* bonds prevail: these polymers show an insulating behavior. On the other hand, when single bonds are alternated with double bonds (*σ* and π), the energy gap is reduced down to the range 1.5-3 eV and thus the molecule exhibits semiconducting properties. The alternation of single and double bonds is called conjugation: in conjugated molecules, *π* bonds are not localized between specific atoms, but alternate between adjacent atoms creating a resonant structure for the electrical conduction in organic molecules. The "doping" of organic semiconductors ,which actually allows the charge transport along the molecule, consists of charge injection by means of dopants, or molecular impurities (halogen vapours in the first experiments carried out by Shirakawa *et al.*), that deform the polymeric chain . The charged particle, together with the geometrical deformation induced, is called *polaron*. This is a quasi-particle with its own effective mass and total momentum energy: in particular, the effective mass is much higher than that of the free charge carriers and therefore its effective mobility is much lower; because of this intrinsic property the effective mobility in organic semiconductors can reach values as high as 10 cm2V¡1s¡1, much lower than 104 cm2V¡1s¡1 of inorganic semiconductors. The polaron induces some atom displacement and its charge is self-trapped by the deformation it has induced: in terms of energy diagram, this effect can be included with an additive localized energy level in the gap between the HOMO and the LUMO of the molecule. Transport between adjacent polymers takes place via ¡ stacking: delocalized orbitals of a molecule overlap the delocalized π orbitals of the adjacent molecule and the polarons can move, with a certain probability, along stacked polymers. The charge transport is strongly influenced by the morphological and structural characteristics of the organic film. While pure inorganic semiconductors are characterized by an ordered crystal structure, organic semiconductors usually have a more disordered, multi-crystalline structure: domains of stacked polymers, called grains, are separated by grain boundaries.

Printed Electronics

Printed Electronics (PE) is a set of materials deposition techniques which literally "print" electronics materials to create a wide range of devices, such as transistors, diodes, detectors, sensors or simple conducting structures and circuits. The main advantages and disadvantages of PE with respect of conventional electronics are *Low fabrication costs, Flexible and transparent substrates, Large areas, Mass production.*

PE technologies are divided in two classes: *contact printing*, deposition of inks by direct contact between the printing plate and the bulk substrate, and *non-contact printing*, by which ink is deposited on the substrate without any contacts with the printing plate.

Contact Printing Techniques

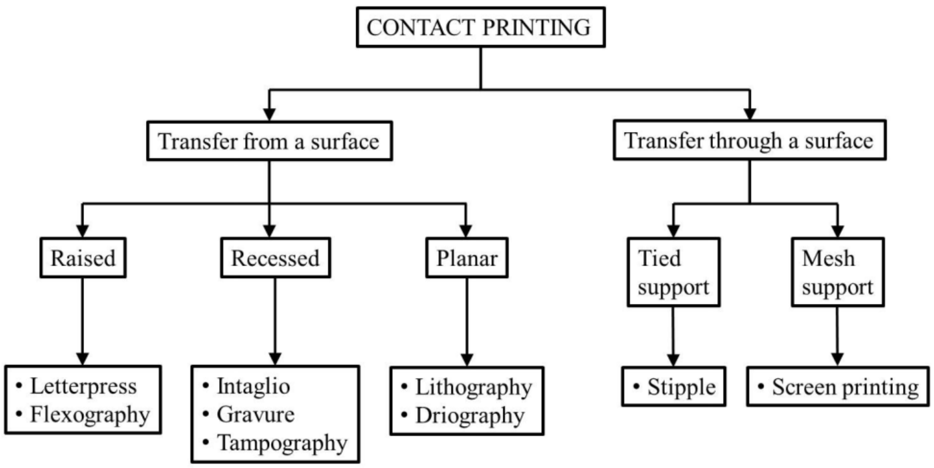


Figure 1: Schematic classification of the main contact printing techniques.

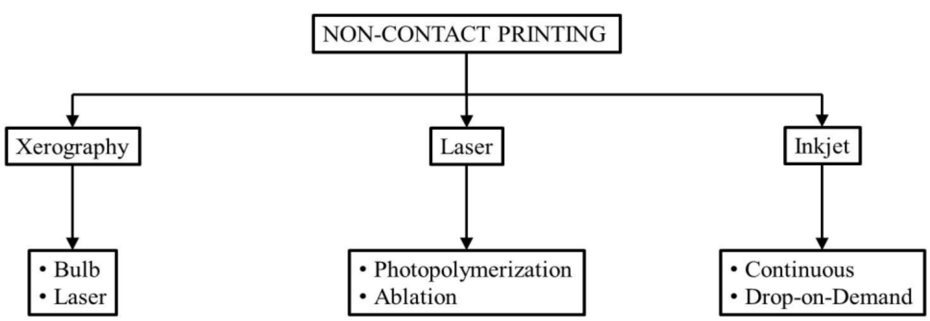


Figure 2: Schematic classification of the main non-contact printing techniques.

INKJET PRINTING

Inkjet printing is a non vacuum process. It mainly consists of the ejection of fixed quantities of ink from a chamber through a hole, called *nozzle*. The ink droplets, under the gravity force, fall on the target substrate and form a patterned layer. An annealing process is then necessary to make the ink solvents evaporate leaving a solid layer on the substrate.

Inkjet printers minimize the material wastage. So in comparison with other techniques of depositing material like chemical vapor deposition, inkjet printer virtually eliminates the issue of wastage of ink. This is an important parameter when printing an expensive material like PEDOT:PSS , MDMO-PPV etc

Unlike other deposition processes which require vacuum to proper functioning, inkjet printing can be performed in normal environment. Because of effective usage of material and cost saved due to non requirement of vacuum for printing the cost of fabricating devices is substantially less as compared to other processes. Further, it proves to be a apt method when it comes to Multilayer deposition which is needed to make a number of devices like Solar Cells. Inks and substrates, conducting, non conducting, insulating, dielectric, resistive etc as well as organic or inorganic in each category, can be printed on a number of substrates. Due to these aspects, inkjet printing of electronic devices offers a unique platform of printing biodegradable organic electronic devices and hence we hose to work with inkjet prnting

.

CHAPTER 2

Inkjet Printers: Classification

Inkjet printers are of two types: Continuous inkjet and Drop on Demand inkjet.

2.1 Continuous Inkjet:

In **continuous inkjet** printing technology, a cylindrical jet of liquid is formed by forcing a fluid under pressure through an orifice. Surface tension creates instabilities in the jet, causing it to break up into drops. By providing a single controlled frequency disturbance, the jet can be forced to break up at precise time intervals into uniform diameter and velocity drops. These can be charged by an electrostatic field at the instant of break-off from the jet, and then deflected from a straight trajectory by a second electrostatic field. Applying multiple levels of charge to the drops allows them to be deflected to one of several locations on a substrate, or to be directed into a catcher for recycling or disposal. Drop sizes varies from 20–150 μm, and even large 1 mm drops (~0.5 pl) have been observed, at generation rates of 80–100 kHz, and up to 1MHz for commercial use.

2.2 Drop on Demand Inkjet:

Drop-on-demand inkjet technology is simpler than continuous inkjet technology and is more widely used. When it is used in combination with piezoelectric print heads, it is the favored process of the printed electronics industry. This technology uses a small transducer to displace the ink, which creates a pressure wave sent to the orifice, where its energy is converted to inertial energy and leads to drop ejection – either an individual or group of drops at random intervals of time, and thus the creation of drops on demand.
Drop size ranges from 15–100 μm (1–500pL) at generation rates of up to 20KHz. Drop-on-demand inkjet systems have no fluid recirculation requirement, but drop generation requires the transducer to deliver at least three orders of magnitude more energy to produce a drop, as compared to the continuous inkjet transducer.

There are two types of DoD inkjet printers:

Thermal Inkjet: drop is generated by heater.

Piezoelectric Inkjet: drop is forced out of the nozzle by pressure pulse.

2.3 Printing Mechanism of piezoelectric DoD Inkjet printer:

Rather than continuous jet of ink coming out of the nozzle, in DoD inkjet printers nozzles are connected to a transducer which converts the electric signal of pulse in physical deformation of the nozzle such that when the nozzle contracts it ejects the ink in the form of droplets and when it expands it sucks in the fresh ink.

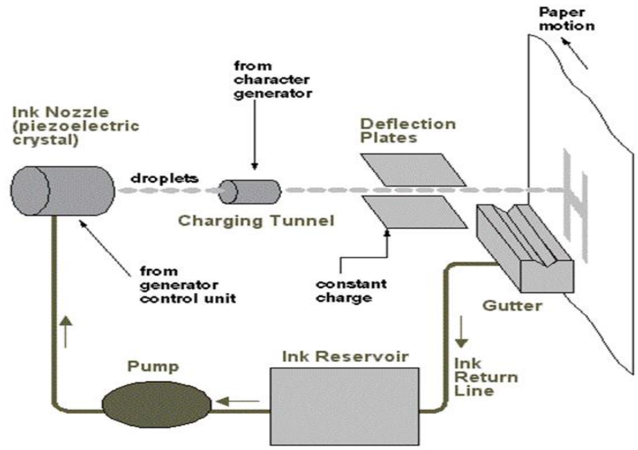


Fig. 3 – Mechanism of DoD InkJet printer

2.4 Printer Specifications:

In our project we have used a Drop on Demand Inkjet printer, Epson L810.
It is a CD-printer, its cost is Rs. 26000/-.
Purchased from amazon.in
Piezoelectric DoD printer.
It is a color printer with 540 nozzles
Therefore, 90 nozzles per color. (6 colors)
Nozzle diameter ~ 20 µm.

Fig. 4– Epson L810, inkjet printer

CHAPTER 3

Substrate

In this project the substrates we ITO(Indium Tin Oxide) coated glass of thickness 24mm, and Glass substrate of thickness 25.4 mm.

3.1 Indium Tin Oxide

ITO glass slides were used as a substrate for fabricating polymer solar cells based on blends of polymeric semiconductors and organic light emitting diodes (OLEDS). ITO substrates can also be used to design transparent heater arrays by etching the surface of the substrate by a printing tool.

3.2 Properties of ITO coated glass:

Refractive Index : 1.8270
Extinction Coefficient : 0.0031008
Relative permittivity : ϵ_1 = 3.3378, ϵ_2 = 0.011330
Absorption coefficient : α = 663.13 cm^{-1}

3.3 Advantages of ITO:

The main concern about ITO is the cost. ITO can be priced several times more highly than aluminium zinc oxide (AZO). AZO is a common choice of transparent conducting oxide (TCO) because of cost and relatively good optical transmission performance in the solar spectrum. However, ITO does consistently defeat AZO in almost every performance category including chemical resistance to moisture. ITO is not affected by moisture and it can survive in a copper indium gallium selenide solar cell for 25–30 years on a rooftop. While the sputtering target or evaporative material that is used to deposit the ITO is significantly more costly than AZO, the amount of material placed on each cell is quite small. Therefore, the cost penalty per cell is quite small too.
The primary advantage of ITO compared to AZO as a transparent conductor is that ITO can be precisely etched into fine patterns. AZO cannot be etched as precisely: It is so sensitive to acid that it tends to get over-etched by an acid treatment . Another benefit of ITO compared to AZO is that if moisture does penetrate, ITO will degrade less than AZO. The role of ITO glass as a cell culture substrate can be extended easily, which opens up new opportunities for studies on growing cells involving electron microscopy and correlative light

Applications of ITO:

ITO is used to make a number of optical coatings and glass substrates. The coatings are used in the manufacture of high expansion glasses, high index glasses and sheet glasses. ITO can offer transparency and conductivity, it is used to coat materials that do not conduct electric current such as plastics. ITO when used in non conductive materials helps to prevent electrostatic charging. Plastic and temperature sensitive substances can also be coated with ITO. ITO can be used forcoatings in industries that manufacture liquid crystal displays, heaters, head-up displays, plasma displays and touch

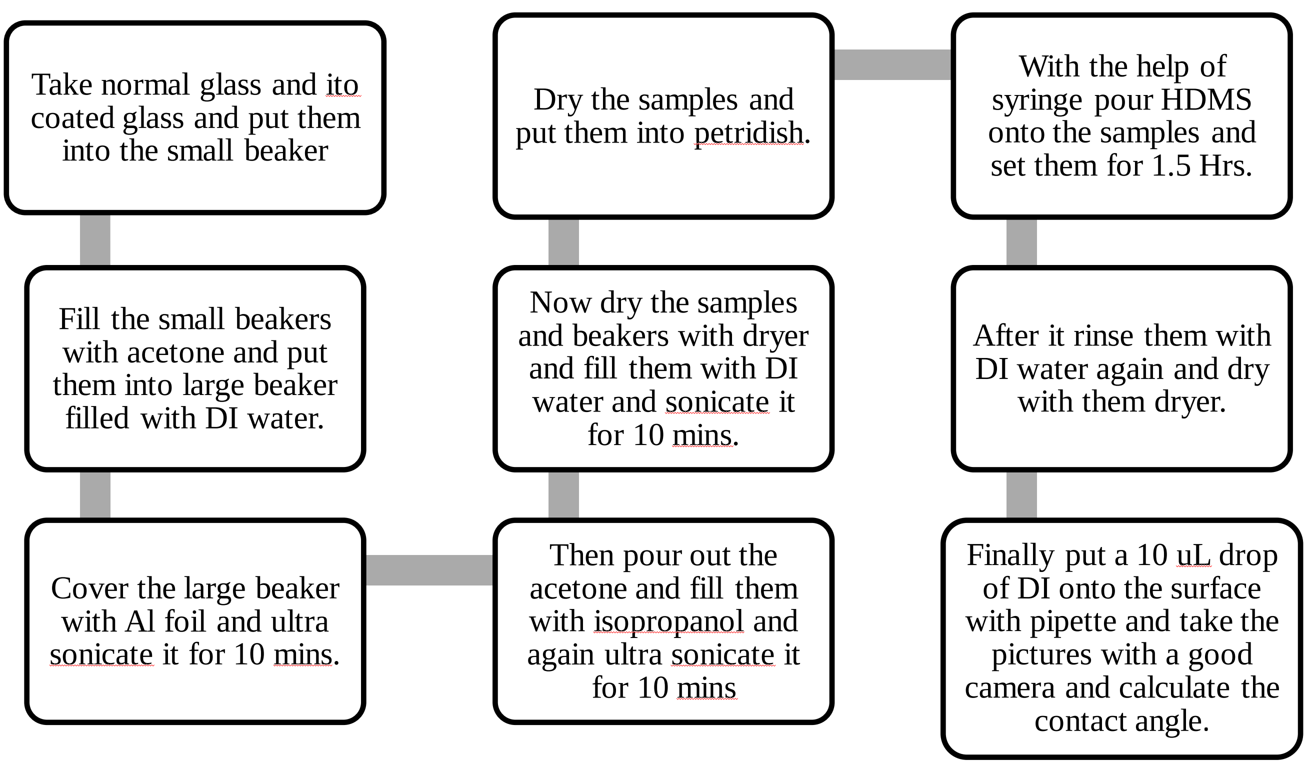
CHAPTER 4

Substrate Treatment

Studies have shown that one of the major factors we came across was wettabilty of the surface, without which the surface may be unevenly coated resulting in the inconsistent properties across the device. One of the indicators of wettabilty is the contact angle, which can be measured using “Imagej” software.

Working on Imagej software

The image is first converted into Greyscale as the software gives accurate results as images in greyscale make it easier for the drop shape to be distinguished. Using the drop snake analysis plug-in the software, we can outline the shape of the drop. The software then checks for different parameters including symmetry and the edge shape among others. The contact angle can thus be measured.

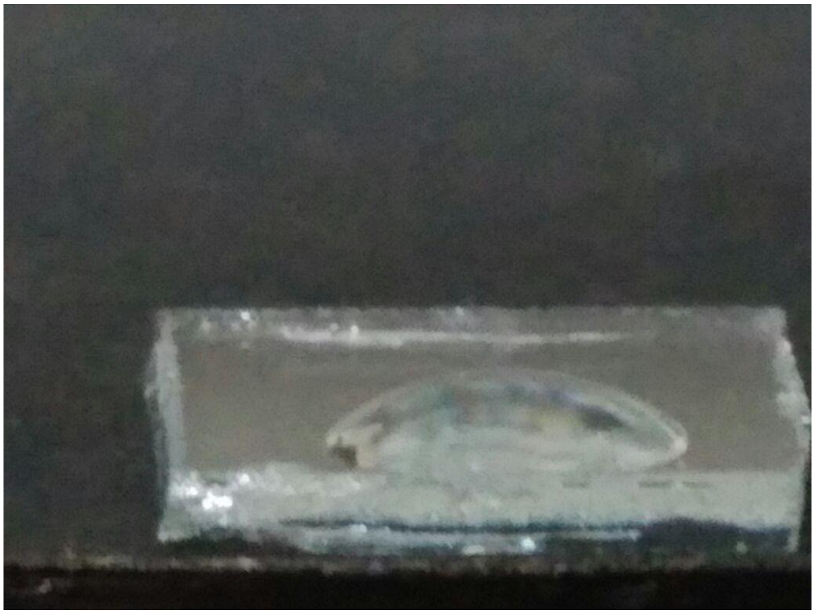


Process involved in the Hydrophobic treatment

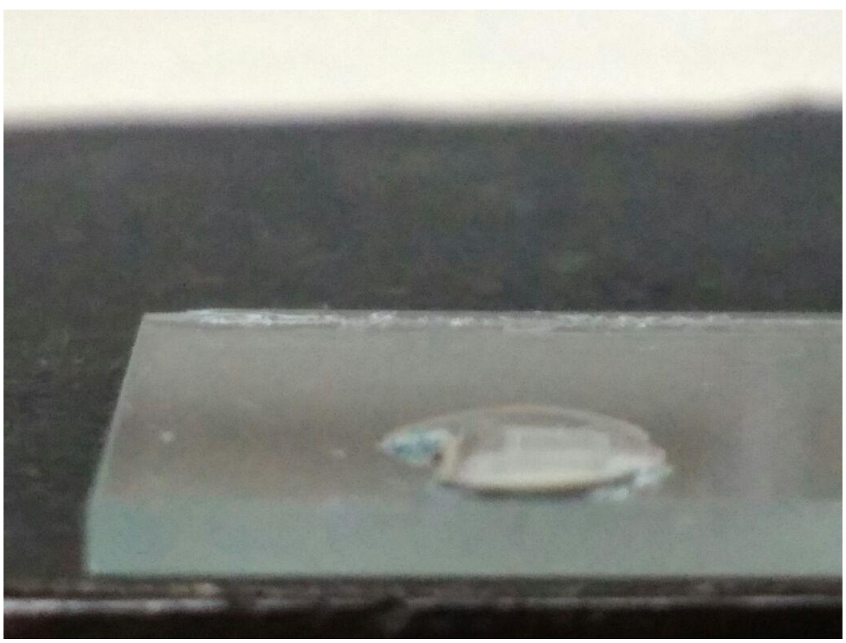
Drop on untreated substrarte (76.6 º)

Drop on treated Substrate(63.5 º)

Similarly we used Pirahna solution for substrate treatment for Hydrophilic nature using standard reagent.



ITO coated glass(36.2º) before treatment



ITO coated glass (25.4º) after treatment

CHAPTER 5

Methodology

Our objective in this project is to print an OLED device using inkjet printing. When printing a device as complicated as an OLED it is important to have the setup optimised i.e. printing parameter of drop spacing , ink composition, surface energy values for the formation of uniform layer. It is equally important to put forward a concept design.

ITO is a p-type transparent semiconductor in the visible range which is often obtained as a degenerated semiconductor. ITO is used as hole-injecting contact that provides positive charges (anode) to the organic layer MDMO-PPV(Poly[2-methoxy-5- (3',7 '- dimethyloctyloxy)-1,4 -phenylenevinylene]). Electroluminescence is produced in the MDMO-PPV film by recombination of electrons and holes.

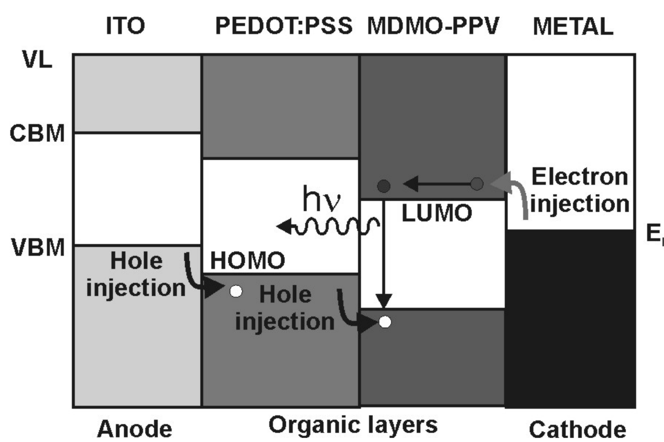


Figure 5: Schematic of OLED

In order to improve the hole injection, a highly conductive and transparent organic layer of Poly(3,4-ethylenedioxythiophene): poly(styrene-sulfonate) (PEDOT:PSS) is used. The transparency of ITO and PEDOT:PSS layers, in the visible range, is a condition needed to avoid absorption of the photons produced in the electroluminescent layer.

As a cathode, several metals could be used for injecting electrons to the organic layer, depending on its work function value. Holes and electrons are injected towards the organic layer from the ITO valence band (EV) and the metal Fermi level (EF), respectively.

Radiative recombination of electron-hole pairs causes electroluminescence of the device. Injection from both electrodes gives rise to bipolar conduction. The barrier for holes (i.e. the energy difference between the valence band of ITO (EV) and the HOMO (highest occupied molecular orbital) in the electroluminescent layer MDMO-PPV) can be effectively reduced by placing an intermediate energy level (HOMO at the PEDOT:PSS layer), which enhances the probability for hole injection.

But before we can print an OLED device we have to make sure that our process is optimised as the costs of PEDOT:PSS and MDMO-PPV are very high. So we decide to fabricate a

Metal/Insulator/Metal structutre of capacitor using poly(4-vinylphenol)PVP
as a dielectric as the cost involved in ink formulation is relatively low.

The device structure for this would be ITO/PVP/Au. PVP would be coated on ITO coated glass and the Au electrode would be deposited by Vacuum Deposition.

CHAPTER 6

Development of MIM capacitor

Formulation of Ink:

PVP is commonly used as a spin-coat dielectric layer for printed field effect transistors and has been demonstrated to be printable. PVP ink has a higher viscosity at a low polymer concentration by weight in the solvent 1-Hexanol which allows for thinner dielectric layers

In order to investigate the versatility of printing thin dielectric films, two PVPh-based dielectric inks are formulated. For simplicity they are referred to as ink1 and ink2. The recipe is composed of the following: poly(4-vinylphenol) (PVPh), a long-chain polymer (Product No. 436216, Sigma Aldrich), poly(melamine-coformaldehyde) (PMF), a heat activated cross-linker (Product No.
418560, Sigma Aldrich), and finally the solvent 1-Hexanol. Ink1 and ink2 have a different ratio by weight of PVPh and PMF, while the ratio between the solvent and the polymers is kept constant to 17:1 w/w%. In particular, ink1 is characterized by 1:10 w/w% ratio while ink2 is formulated with 1:1 w/w% ratio, between PVPh and PMF. Both PVPh and PMF are polymers with dielectric properties, so changing their ratio allows for the tuning of the permittivity (ε) and the loss tangent (tand) of the printed thin film layer.

Ink	PVP	PMF	Hexanol
1	5%	0.5%	94.5%
2	2.75%	2.75%	94.5%

Table 1:Recipes (ratio by weight) for the PVPh-based ink variations

Problems faced and rectifications:

The ink1 used clogged the head of the printer(radius 10µm) despite the process of ink formulation being in accordance with the parameter of inkjet printing. The second ink though showed printing results, due to agglomeration the head was clogged again.

Method used to clean the printer:

Head of the printer was cleaned repeatedly with Acetone, Ethanol and IPA as only the organic solvents are capable of removing agglomerated particles.

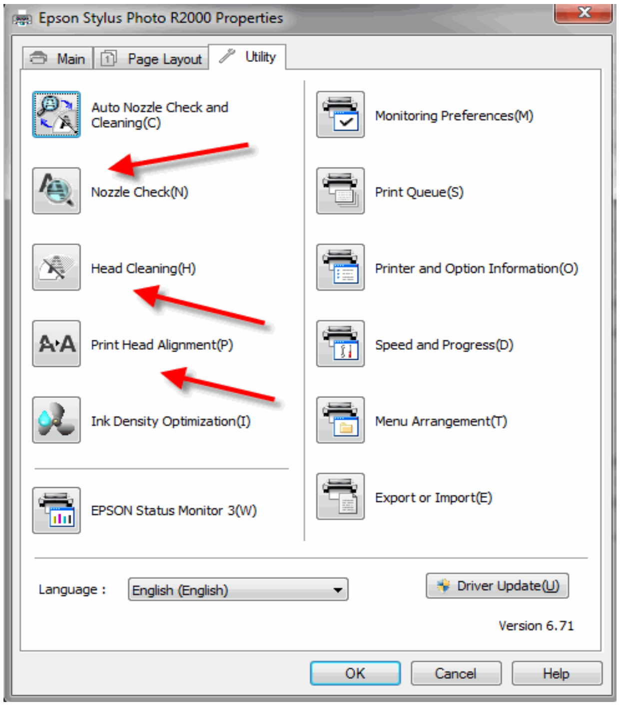


Figure 6:Head cleaning using software

Figure 7:Head cleaning with VMS cleaning

We tried to reduced the viscosity and increased the surface tension by adding ethanol as solvent because the printer kept getting clogged. This resulted in very poor quality of the films.

Due to repeated coagulation and blocking of the nozzle of the printer the mother board of the printer was short circuited.

Obtaining a printer was not feasible so we switched from Inkjet printing to Doctor Blading.

CHAPTER 7

DOCTOR BLADING

Doctor blading is a technique where the ink of the material to be coated is deposited in front of the doctor blades and the material is literally coated using a blade by moving the blade along the substrate. The main advantages of Doctor blading are that the starting material is very low. The process can be easily transferred to R-2-R manufacturing. It can be used for a large area of fabrication with negligible cost. The working temperature depends on the inks which can be adjusted to room temperature. The thickness of the layer printed depends upon the gap between the blade and the substrate.

The parameters to focus while using Doctor blading were that we the uniformity of the blade over the surface and stability of the blade. To ensure this we made a film applicator in which channels with rollers were used to ensure that there is no horizontal movement . and the height of the blade over the surface(i.e. the thickness of the film) can be varied by adjusting the channel position.

Figure 8: Film Applicator channels

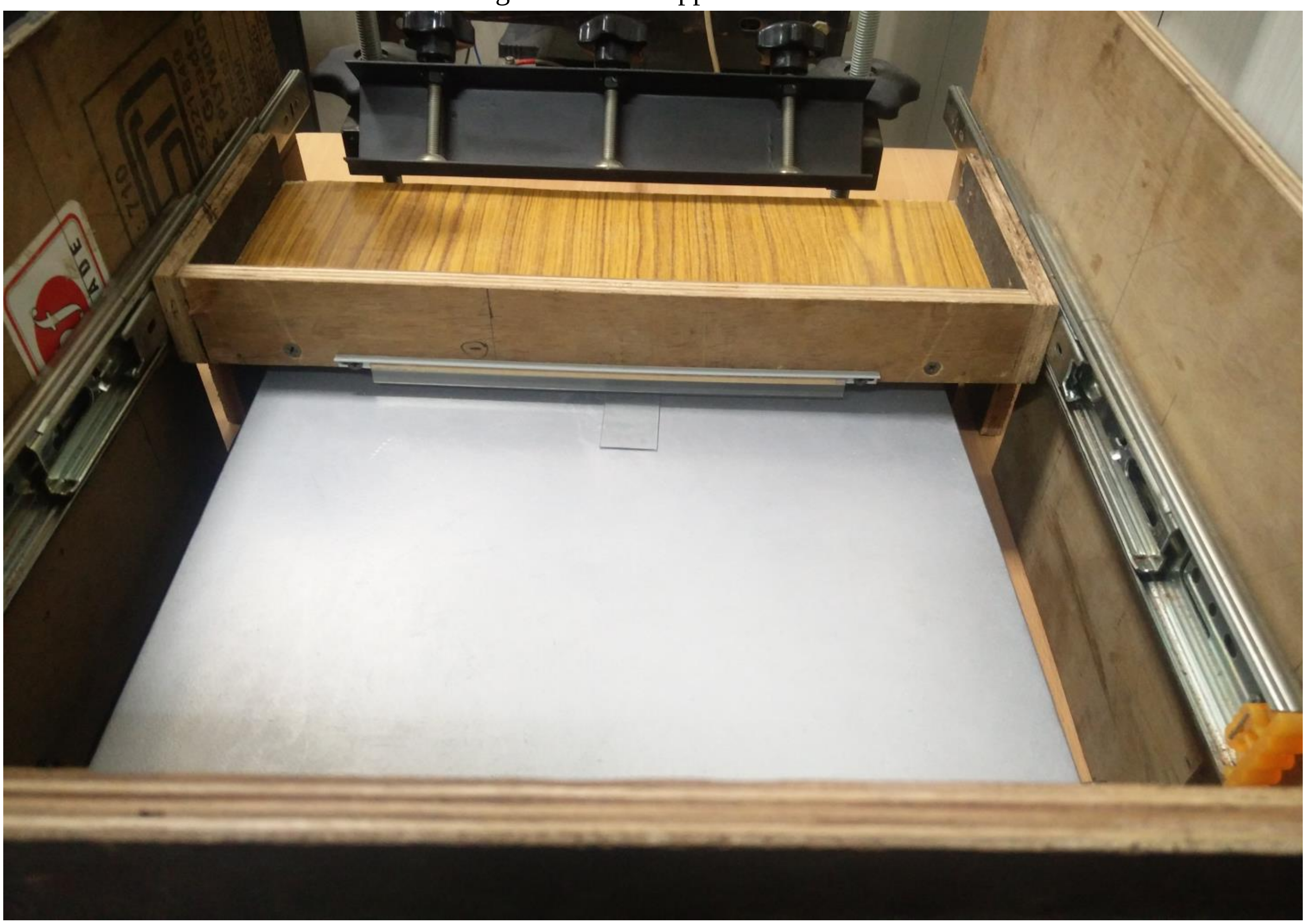


Figure 9:Front view of the film Applicator showing the doctor blade

FABRICATION OF MIM CAPACITOR

For reference, we have fabricated the capacitor via Spin Coating. The capacitor with composition ITO/PVP /Au was fabricated. The thickness of the Au was kept 50µm .

ITO coated glass was used as substrate.

As the viscosity needed for doctor balding is more than that of the inkjet printing, Ink with 15%wt of PVP was formed with PGMEA as solvent. The rotational speed was kept [1.5,3] Krpm.

The Au electrode was Vacuum Deposited after drying.

CHAPTER 8

Results and Conclusion

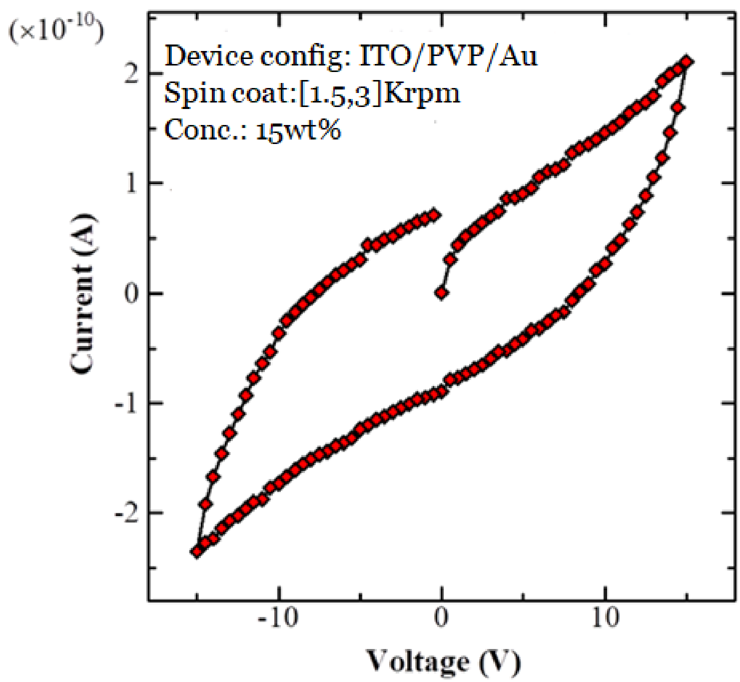


Figure 10:I-V charachteristics of capacitor

Cyclic Voltammetry graph of capacitor ITO/PVP/Au

Cyclic Voltammetry analysis and frequency variations were done using Keithley 2612b source-meter.

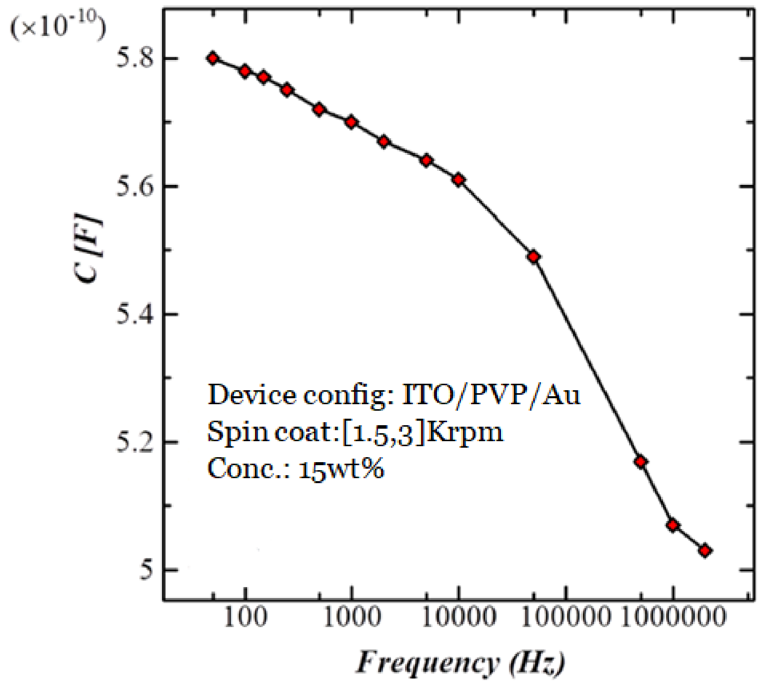


Figure 11:Capacitance v/s Frequency of ITO/PVP/Au

Conclusion:

1. Agglomeration is one of the biggest challenge while preparing Organic inks,

Following data can be used to solve this problem

Colour	Surface Tension (mN/m)	Viscosity(mPa.s)
Black	44.3	1.85
Red	33.8	1.95
Yellow	39.4	1.85
Blue	34.2	1.95

Table2: Data of Epson inks

2. Doctor Blading Technique may generate pinholes due to insufficient wetting and non uniform speed.
3. Automation of doctor blading film applicator will produce required thin films with less cost of production.

CHAPTER 9

FUTURE SCOPE

Optimization of Film Applicator and it's automation to print multilayer structure of OLED.

Continuing the work on inkjet printer and optimizing the printing parameters of drop spacing, nozzle velocity and input voltage.

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