B.TECH. PROJECT REPORT

On

Deposition of ZnO thin films on flexible polyimide substrate using ink jet printer

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DISCIPLINE OF MECHANICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY INDORE,

November 2016

Deposition of ZnO thin films on flexible polyimide substrate using ink jet printer

A PROJECT REPORT

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

of

BACHELOR OF TECHNOLOGY

in

MECHANICAL ENGINEERING

Submitted by:

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Guided by:

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INDIAN INSTITUTE OF TECHNOLOGY INDORE, November 2016

CANDIDATE'S DECLARATION

We hereby declare that the project entitled **"Printing of semiconductor and polymers on flexible polyimide substrate, using inkjet printer"** submitted in partial fulfillment for the award of the degree of Bachelor of Technology in MECHANICAL ENGINEERING completed under the supervision of **Dr. I.A. PALANI** [ASSOCIATE PROFESSOR] and **Dr. Vipul Singh**[ASSOCIATE PROFESSOR]**,** IIT Indore is an authentic work.

Further, I/we declare that I/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 Guide(s)

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

Signature of BTP Guide with dates and their designation

Preface

*This project report on **"Deposition of ZnO thin films on flexible polyimide substrate using an ink jet printer"** is prepared under the guidance of Dr.I.A.Palani & Dr. Vipul Singh.*

In this report we have discussed two methods and their pros and cons for depositing ZnO on polyimide substrate with soul aim to make a contribution to one of the most intriguing technology developing nowadays, printed electronics using inkjet printer.

We have tried our best to explain this content to a reader in a lucid manner with comprehensive theory. We have added photographs and experimental results to make it more illustrative.

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Acknowledgements

We wish to thank **Dr. I.A.Palani** & **Dr. Vipul Singh** for their kind support and valuable guidance. This work has been done in framework of the IIT Indore project that is funded by the **PRIUS**(Promotion of Research & Innovation for Undergraduate Student). We would like to acknowledge **Mr. Ashish Shukla** and **Mr. P. Rajagopalan**, Mechatronics & Instrumentation lab, Chemistry Lab, Molecular and Nanoelectronics Research Group(Dept. of Electrical Engg.), Central Workshop and Sophisticated Instrumentation Centre of IIT Indore for providing their sincere cooperation and guidance to carry out this research.

It is their help and support, due to which we were able to complete the design and the technical report. Without their support this would not have been possible.

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Abstract

In this article, ZnO thin films deposited on flexible polyimide substrate by inkjet printing approach are studied. For this purpose, firstly an intermediate aqueous solution of Zinc acetate dihydrate with monoethanolamine as stabilizer was inkjet printed on substrate. After this we synthesized ZnO nanoparticle ink in organic solvents. ZnO NPs were dispersed in hexylamine before adding to, and ethylene glycol, isopropanol, toluene (mixed in weight ratio of 0.05:0.1:0.8), because first ink was unstable due to agglomeration. This ZnO nanoparticle ink was deposited on polyimide substrate through inkjet printing. The particle size in ZnO nanoparticle ink was measured by dynamic light scattering and it was found to be about 275.48 nm. Polyimide surface was pretreated with aqueous solution of KOH for about one hour to improve adhesion. Printed substrates were studied by X-ray diffraction, scanning electron microscopy, photoluminescence. Further, the adhesion was tested by scotch tape adhesion test.

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Chapter 1
Introduction

Electronic circuits or devices are the foundation of the present day technology. All the electrical equipments, for their effective and efficient functioning, rely on these electronic parts. Electronics devices have been fabricated on rigid components like silicon wafers traditionally. Recently, making devices on flexible substrates have gained a lot attention and there has been a major shift in focus of the electronic device makers and giant electrical organizations from so called 'traditional rigid electronics', to 'printed electronics' or more specifically, known by the name '**flexible electronics**'.

Flexible electronics typically involves a number of different techniques, employed to fabricate flexible electronic devices such as flexible organic light emitting diodes (OLEDs), flexible solar cells, flexible substrates in LCDs etc.

The **advantages** of flexible electronics over its conventional counterpart can be identified from:

They are ***Light:*** Flexible electronics can replace wire harness (which are bulkier and heavier) in Rockets and Satellites where weight of machine matters a lot.

They are ***flexible***: can be used in making folding cell phones and cameras.

They are ***thin:*** can be used in applications where thick and rigid boards pose problems.

Also, due to above characteristics transportation of these devices becomes easier.

Inkjet Printing, Flexography, Gravure printing etc. are some of the techniques used for fabricating flexible electronic devices.

In traditional device fabrication, first an organization manufactures silicon wafers, modifies its surface properties, and then passes the wafers through phosphorous baths in order to get p-n junction finally using screen printing or lithography techniques to deposit Aluminium or Silver to make conductive circuits, all these steps make the devices costly.

Whereas, here inks are formed which can be conducting, insulating, semiconducting in nature or may be dielectric. These inks are fed into the printer and deposited on number of substrate. Further pretreatment or post treatments can be done to improve other properties like adhesion of inks onto the substrates.

In this project, we have done experiments on adhesive properties of polyimide (thickness 50 µm, kapton) also increasing degree of its hydrophilic nature. Further, a lot of study was done and different materials were studied to make an ink. We aimed at depositing Zinc Oxide on flexible polyimide substrate using an Epson L810 inkjet printer. Two different methods were used to deposit ZnO on polyimide. In first method an intermediate solution was formed, after depositing the solution, polyimide was annealed to a temperature of 380^{o} C, after annealing solvent evaporates leaving behind ZnO on substrate. In second method ZnO nanoparticles were synthesized, they were dispersed hexyl amine and mixed with organic solvents and filtered with 0.45µm syringe filter to make ZnO nanoparticle ink. Resulting ink had the average particle size of around 275.48 nm measured with DLS particle size analyzer. Further several characterization tests were done to confirm the presence of ZnO nanoparticles.

Why Inkjet printing?

The most eye-catching advantage of inkjet printing over other methods is that it is user friendly. Printing procedure is so easy that anyone can perform it. Even complicated circuit design, can be easily designed in PC and printer can be ordered to deposit the same pattern. Another key aspect is its accuracy. Inkjet printing is highly accurate in positioning of the ink droplets and fully efficient in creating every minute detail of designed pattern (in PC) on the substrate. Since the computer's commands are generated from the software, updates can be made in real-time to the patterns. This is in contrast to gravure printing where the patterns are engraved into rollers. So, if you have to make changes in pattern design a gravure printing technique would require a new pattern to be mounted on rollers which is both time and cost consuming, whereas in inkjet printing a small adjustment to design of in your PC will start printing accordingly on the next substrate without any such complications.

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 Silver or CIGS etc.

Inkjet printing is a non vacuum process. 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 powerful method when it comes to Multilayer deposition which is needed to make a number of devices like Solar Cells.

Moreover, inkjet printing offers variety. A good number of inks and substrates either 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.**

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 favoured 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.

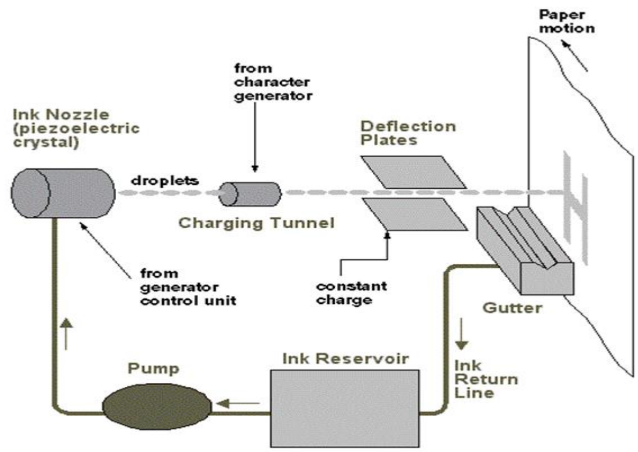

Fig. 1 – Mechanism of DoD InkJet printer

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. 2 – Epson L810, inkjet printer

Chapter 3
Substrate

In this project polyimide sheet of thickness 50 μm was used as the substrate. Other substrates were also used like OHP sheet and Inkjet Printable CDs.

3.1 Polyimide

Polyimide (sometimes abbreviated PI) is a polymer of imide monomers. Polyimides have been in mass production since 1955. With their high heat-resistance, polyimides enjoy diverse applications in roles demanding rugged organic materials, e.g. high temperature fuel cells, displays, and various military roles. A classic polyimide is Kapton, which is produced by condensation of pyromellitic dianhydride and 4,4'-oxydianiline.

3.2 Properties of polyimide

Polyimide offers an excellent combination of electrical properties, thermal conductivity and mechanical toughness for its use in electronic and automotive applications.

Some of the electrical properties of polyimide are:

Dielectric Strength	5500 V/mil
Dielectric Constant	4.2
Thermal Conductivity	0.46 W/m-K
Volume Resistivity	$>10^{16}$

Thermosetting polyimides are known for thermal stability, good chemical resistance, excellent mechanical properties, and characteristic orange/yellow color. Polyimides compounded with graphite or glass fiber reinforcements have flexural strengths of up to 340 MPa and flexural moduli of 21,000 MPa. Thermoset polyimides exhibit very low creep and high tensile strength. These properties are maintained during continuous use to temperatures of up to 452 °C and for short excursions, as high as 704 °C.

Polyimide film is made by a condensation reaction; therefore, its properties are affected by water. Although long-term exposure to boiling water, will reduce the level of film properties, sufficient tensile and elongation remain to ensure good mechanical performance. A decrease in the temperature and the water content will reduce the rate of property reduction, whereas higher temperature and pressure will increase it.

3.3 Advantages and Applications of polyimide

Since polyimide materials are chemically resistant, i.e., they are not affected by solvents and oils. In addition to this, they also pose a resistance to mild acids but are not recommended for use in environments that contain alkalis or inorganic acids.
Polyimide materials are **lightweight, flexible, resistant to heat and chemicals.** Therefore, they are used in the **electronics** industry for flexible cables, as an insulating film on magnet wire and for medical tubing. For example, in a laptop computer, the cable that connects the main logic board to the display (which must flex every time the laptop is opened or closed) is often a polyimide base with copper conductors.

An additional use of polyimide resin is as an insulating and passivation layer in the manufacture of digital semiconductor and MEMS chips. The **polyimide layers have good mechanical elongation and tensile strength**, which also helps the adhesion between the polyimide layers or between polyimide layer and deposited metal layer.

CHAPTER - 4
Substrate treatment

Polyimide is hydrophobic in nature. To increase the hydrophilicity of polyimide, in order to print water-based inks on its surface, surface treatments can be done. One such surface treatment is treating the polyimide surface with aqueous solution of potassium hydroxide (KOH).
When polyimide reacts with potassium hydroxide solution, the imide ring opens allowing water based inks to stick to the surface effectively owing to the similar surface energy level of surface and water based cause by KOH treatment.

4.1 Reactions:

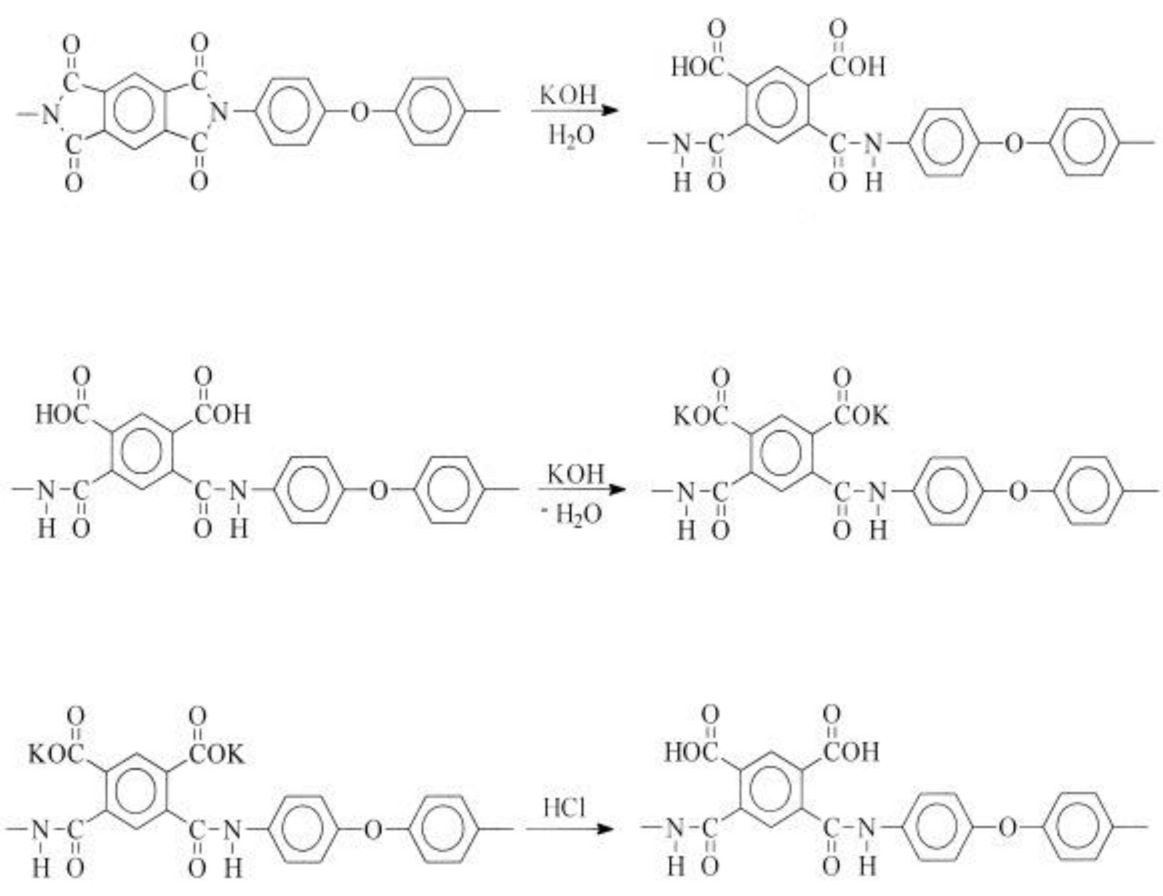

Fig. 3 – Reaction of polyimide with KOH solution

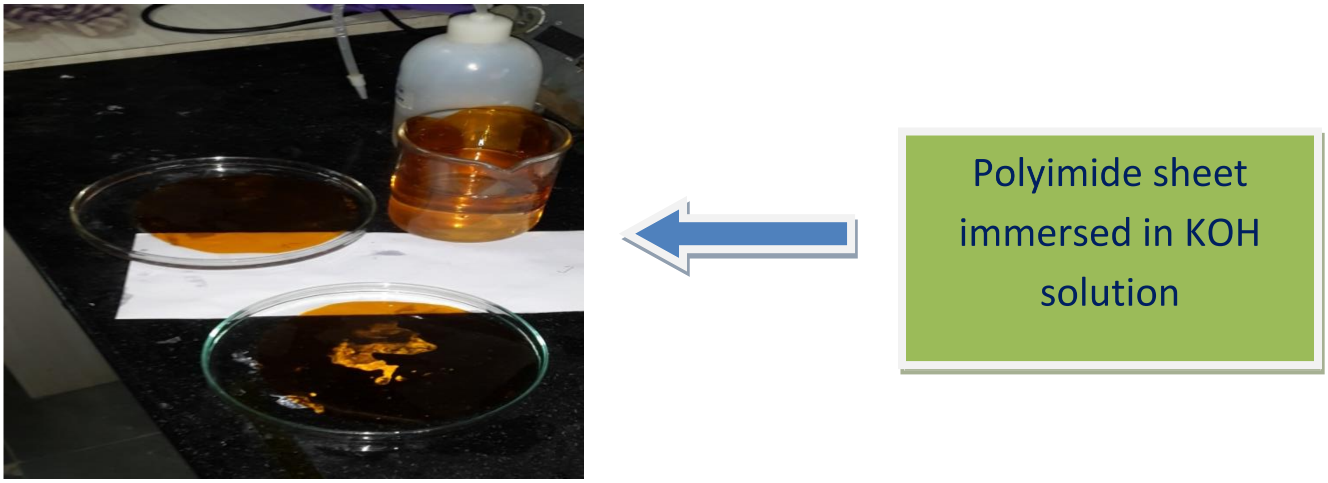

Fig. 4 – Surface Treatment

Polyimide sheets were cut, and ultrasonicated with isopropyl alcohol and distilled water for 10 minutes with each reagent. After this they were immersed in KOH solution for about one hour. Normal inkjet ink was printed on Polyimide sheets, both KOH treated sheet and Untreated sheet.

Untreated **Treated**

Fig. 5 – Effects of Surface Treatment

It can be clearly observed that the print quality of water based inkjet ink increases after treating polyimide sheet with KOH, due to better adhesion between the ink and the substrate.

Chapter 5
Deposition Of ZnO on polyimide

5.1 Using intermediate solution of Zinc acetate dihydrate

When Zinc acetate dihydrate is dissolved in water and monoethanolamine it forms an intermediate solution in which Zinc is present. This solution when deposited on a substrate gets adhered to the surface.
In the next step the substrate is annealed to 300-400 ^{o}C temperature, due to this heat intermediate Zinc compound gets converted to ZnO (Zinc Oxide) and solvent gets evaporated.

In this method, a solution was formed Zinc Acetate Dihydrate as the main component. The Zn precursor solution was prepared by dissolving 0.01 mol of $Zn(CH_3COO)_2.2H_2O$ (Sigma Aldrich, 99%) in 10-mL water. Zinc acetate dihydrate has a low solubility in water, and the reaction is slow. To increase the rate of the reaction and solubility of Zinc acetate dihydrate, monoethnanolamine was mixed with water till a clear colorless solution was obtained. Formic acid was then added to the solution till the pH of resulting solution was equal to 7. The printability of the ink was improved by the addition of 10 v% ethanol. At the end, a clear 0.5 mol/L Zn precursor was achieved by diluting the precursor solution with water.

The synthesized solution was used as an ink for printing with Epson L810 inkjet printer. The substrate used was Inkjet printable CD – Philips (amazon.in).

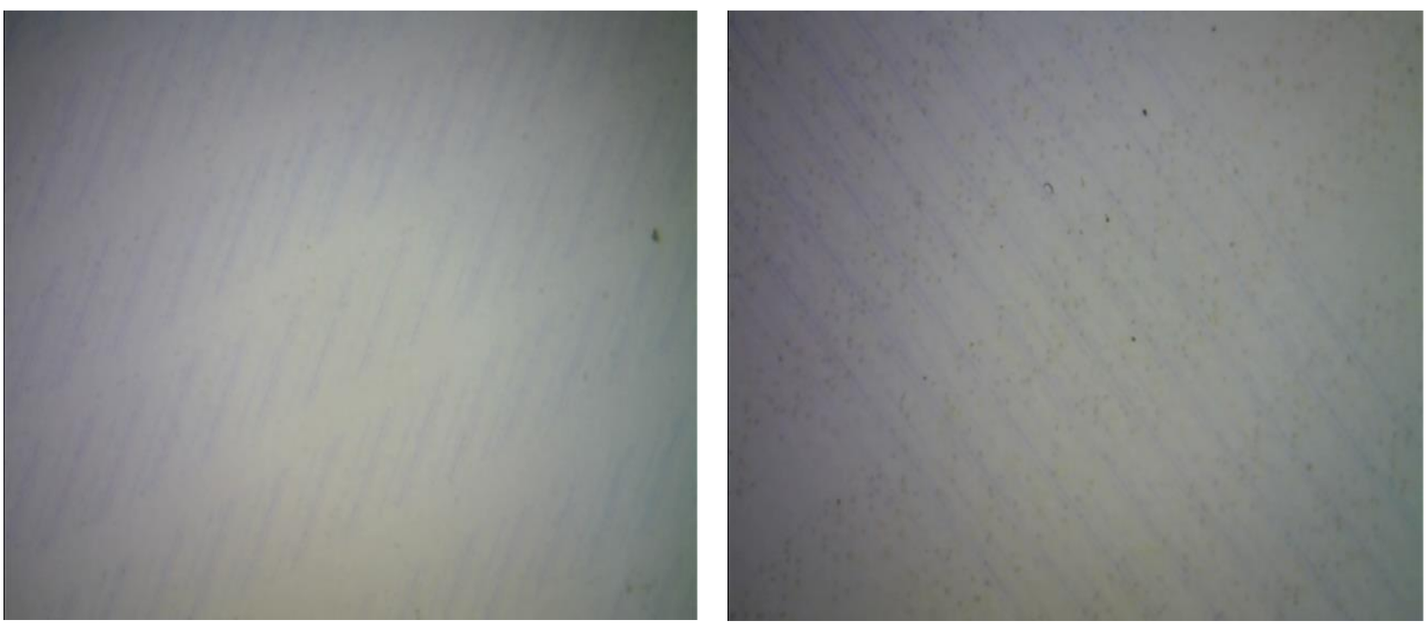

Fig. 6 - Images of Line patterns printed on Inkjet printable CD taken by USB digital microscope

5.2 Problems Faced and Rectifications

The patterns were successfully printed on inkjet printable CD. Next, printing on our main substrate, i.e. polyimide, was yet to be tested. After giving the print command we noticed that no deposition was happening. On investigation we found that the nozzles had clogged. The Zinc intermediate solutions appeared clear at first but with time due to agglomeration, the particles got bigger in size, thereby their size becoming larger than the nozzle diameter of 20µm. Because of this the nozzles got clogged.

To clean the nozzles, different solutions with low viscosity were made to react with Zinc acetate dihydrate. We observed that this particular Zinc salt was readily soluble in IPA (Isopropyl alcohol). To clean the nozzles mild pressures were applied with syringe filled with IPA. This solved was the problem up to some extent but still some of the nozzles were not unclogged. Now, a cleaning solution was purchased and nozzles in the print head were dipped in it and the solution was sucked from the nozzles with the help of the 1 ml syringe.
After repeating these processes several times the nozzles were fully functional once again.

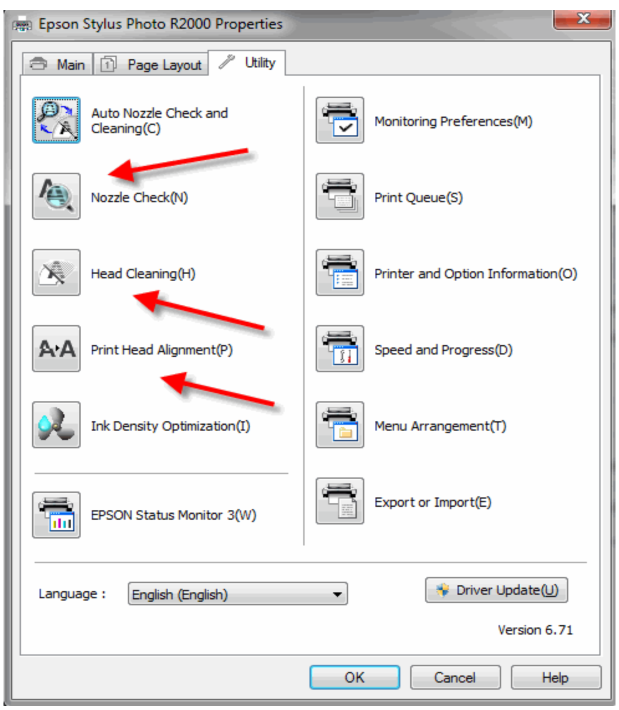

Fig. 7- Head cleaning from software

Fig. 8 - Cleaning Print head with VMS cleaning

Chapter 6
ZnO Nanoparticle Ink

The solution obtained was not stable because of agglomeration of small particles to form larger particles. Learning from the previous experience we shifted our focus to make ZnO nanoparticles and dispersing these nanoparticles in an organic solvent to form a stable organic ink.

6.1 Advantages of Nanoparticle ink:

- Lower particle size eliminating the problem of nozzle clogging.
- Stable suspension: leading to sustenance of ink properties for longer time.
- Desirable surface tension.
- Uniform dispersion of particles.

6.2 Synthesis of ZnO Nanoparticles

To synthesize ZnO nanoparticles, 0.1 M anhydrous Zinc Acetate solution was prepared in 200 mL aqueous solution and then NH_4OH was added to adjust pH=10 of the solution under stirring at 40–50 ^{o}C. After adjusting the pH, the solution was ultrasonicated at room-temperature for 1 hour in an ultrasonicator (20 kHz). To check the effect of ultrasonication on the purity and crystallinity of ZnO nanoparticles, we performed experiments based on with and without ultrasonication. As-obtained white colored colloidal suspension were centrifuged at 3000 rpm for 5 min, washed with ethanol several times, and dried at room-temperature.

Fig. 9 - Measurement of exact amount of Zinc Acetate with high precision digital balance

Fig. 10 - Stirring of solution of Zinc Acetate with water

6.3 Calculation

0.1 M solution of Zinc Acetate was formed in 200 mL water.
Molecular Weight of $Zn(AC)_2$ = 183.48 gm/mol
So, quantity of Zinc Acetate used was

0.1 x 200/1000 x 183.48 = **3.66** gm

Ethylene Glycol, Isopropanol, Toluene were added in weight ratio of 0.05:0.1:0.8.
So, to calculate volume ratio, we need to divide by density.

Ethylene Glycol = 0.05/1.11 = 0.045

Isopropanol = 0.1/0.786 = 0.127

Toluene = 0.8/0.867 = 0.9227

Hexylamine = 0.05/0.77 = 0.065

Hence,

Components	Volume Percentage in ink	Amount for making 40 mL ink (in mL)
Ethylene Glycol	4	1.6
Isopropanol	11	4.4
Toluene	80	32
Hexylamine	5	2

6.4 Ink-Formulation Using Synthesized ZnO nanoparticles

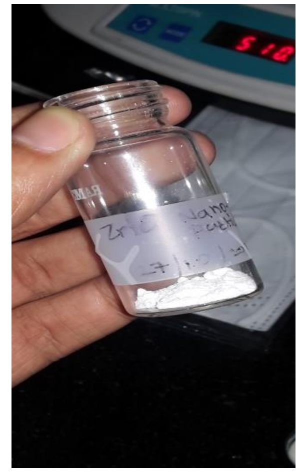

Fig. 11 - Nanoparticles of Zinc Oxide

ZnO nanoparticles inks were prepared in a mixed solvent of ethylene glycol, isopropanol and toluene. Hexylamine was used as a dispersing agent to enhance the dispersion of ZnO nanoparticles in the mixed solvent. In a typical ink-formulation, firstly 0.2 g of as-synthesized ZnO nanoparticles was mixed in 4mL Hexylamine solution and stirred for 12 h. After this process, ethylene glycol, isopropanol and toluene were mixed with weightratio of 0.05:0.1:0.8. The formulated ZnO ink was then filtered by 0.45 µm filter paper before jetting. The ZnO pattern lines were printed on Polyimide and OHP substrates.

6.5 Equations Involved:

$Zn(AC)_2 + 2\ NH_4OH \rightarrow Zn(OH)_2 + 2\ NH_4AC$

$Zn(OH)_2 \rightarrow ZnO + H_2O$ (on heating at 350 °C)

6.6 Printing Setup:

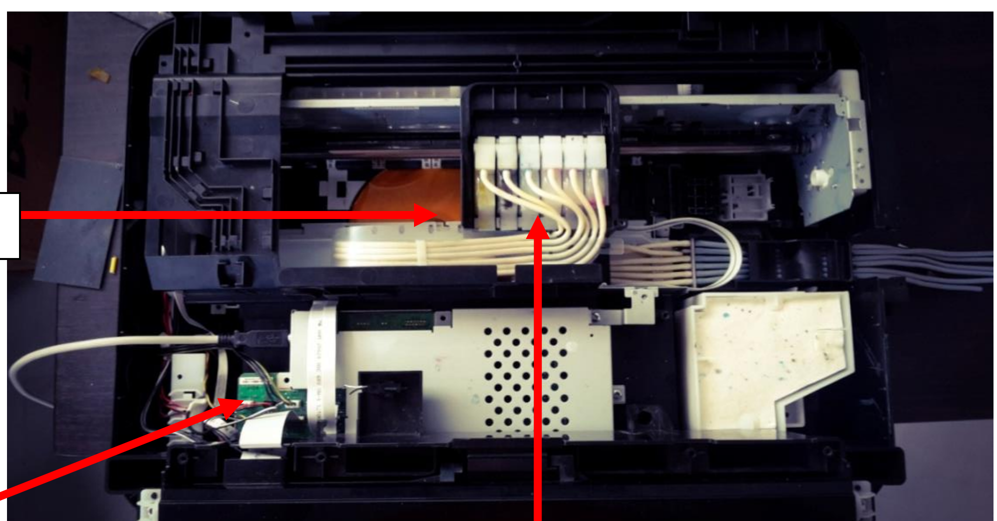

Fig. 12 – Printing Setup

Chapter 7
Images of ZnO printed patterns

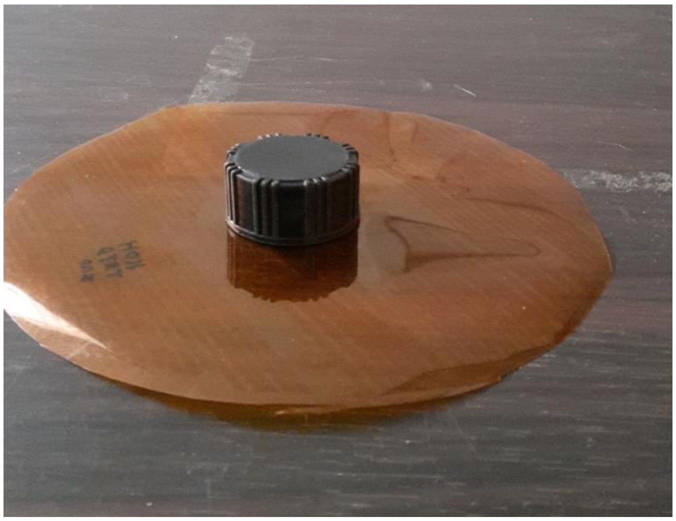

Fig. 13 - ZnO lines on PI

Fig. 14 - ZnO lines on OHP

Fig. 15 – ZnO pattern printed on PI

Fig. 16 - Magnified image of printed PI sheet taken by USB Digital

Chapter 8
Applications of ZnO

ZnO is a wide-band gap semiconductor of the II-VI semiconductor group. The native doping of the semiconductor due to oxygen vacancies or zinc interstitials is n-type. This semiconductor has several favorable properties, including good transparency, high electron mobility, wide band gap, and strong room-temperature luminescence.

Those properties are valuable in emerging applications for:

- Transparent electrodes in liquid crystal displays,
- Energy-saving or heat-protecting windows, and
- Electronics as thin-film transistors
- Light-emitting diodes.
- UV photo detectors
- Solar Cells
- Gas Sensors
- Humidity Sensors
- Energy Harvesting Etc.

Chapter 9
Results and Discussion

After the synthesis, the ink was put to test for printing. Different substrate such as polyimide, OHP sheet and Inkjet printable CDs were used. On all the substrates used the print quality observed was good.
To check the presence of Zinc Oxide particles on the substrates several tests were performed.

9.1 X- Ray Diffraction:

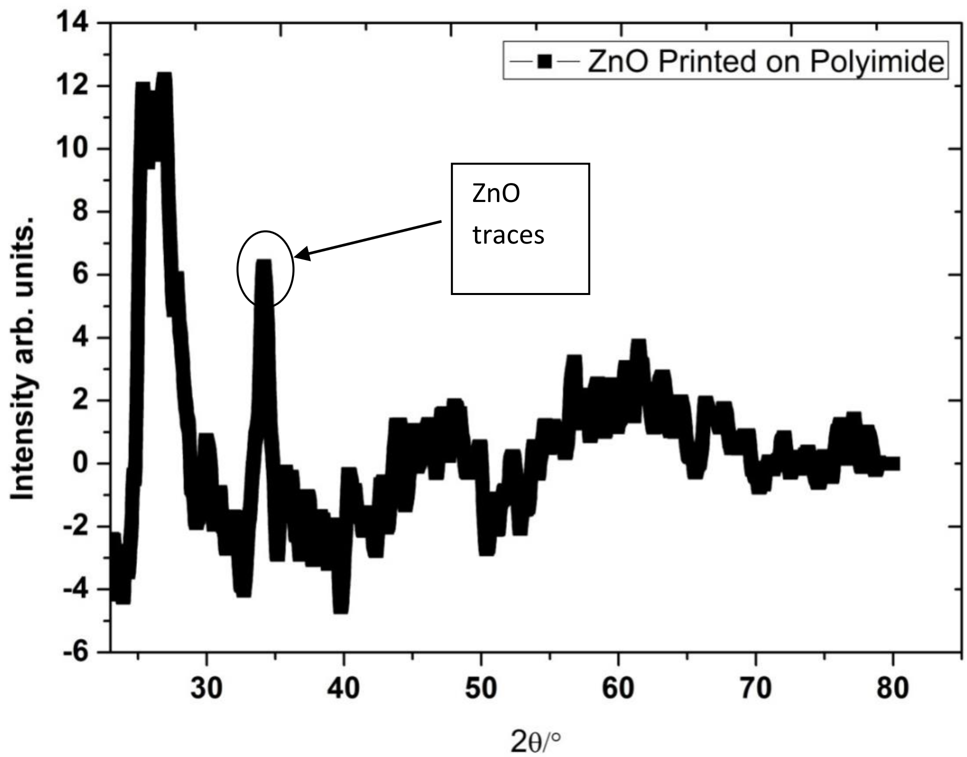

Fig. 17 – XRD of ZnO deposited on PI sheet

X-ray diffraction of polyimide sheet on which synthesized ZnO ink was inkjet printed was done. The zigzag nature of graph is because of the amorphous nature of polyimide sheet which results in non uniform diffraction due to random arrangement of molecules. The peak between 30-40 degrees confirms the presence of ZnO particle on the substrate. This behavior of graph can be attributed to thin layer of ZnO deposited on polyimide.

9.2 DLS – Dynamic light scattering:

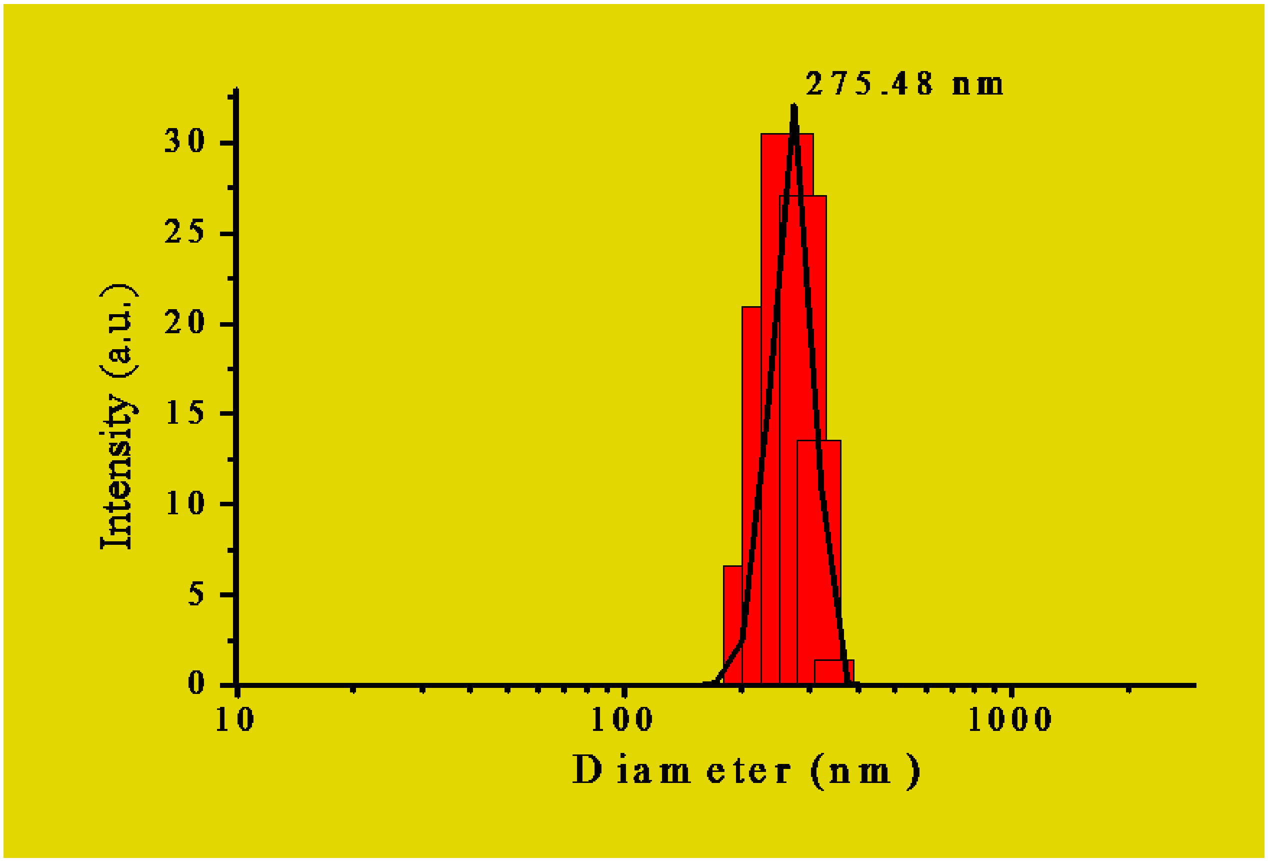

Fig. 18 – DLS Particle Size Analyzer Data

DLS – Dynamic Light Analyzer was used to measure the particle size of ZnO nanoparticles dispersed in toluene. The average particle size was found to be 275.48 nm.

9.3 Photoluminescence:

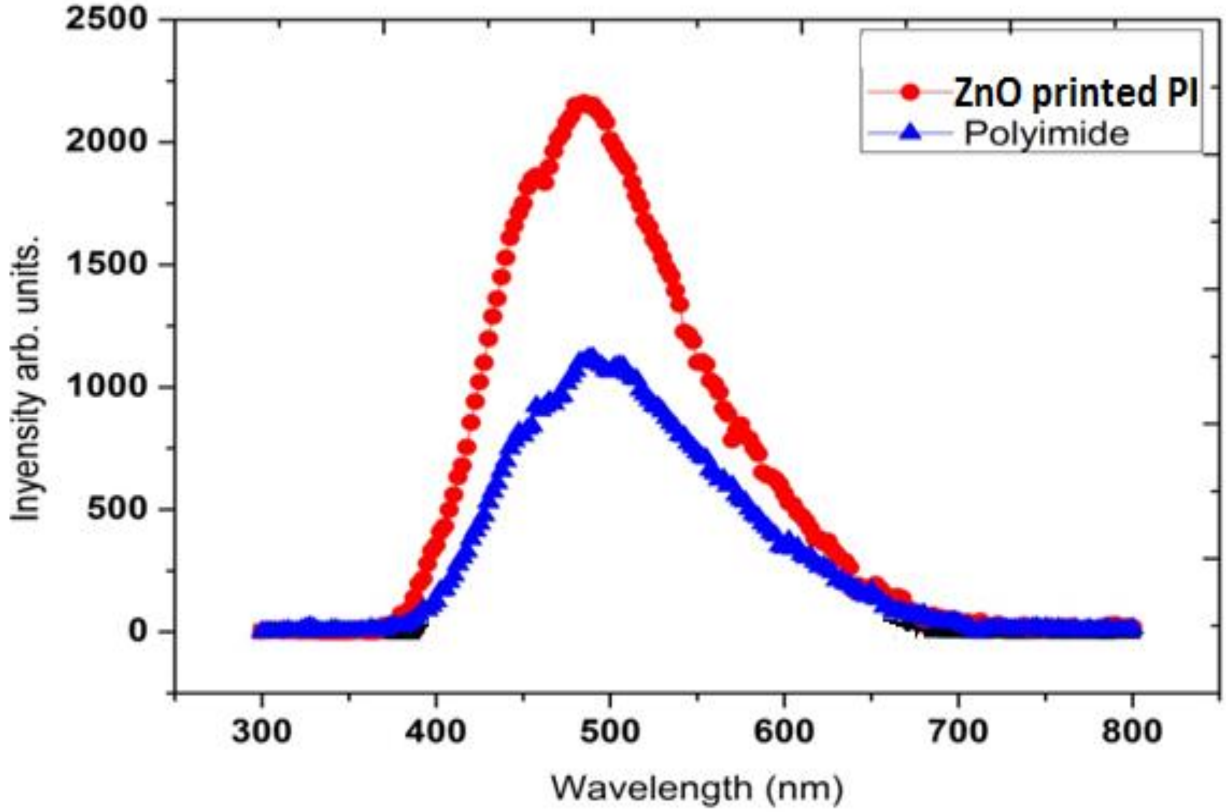

Fig. 19 – Photoluminescence graph of ZnO on PI

Photoluminescence of the ZnO printed polyimide substrate gives a large characteristic peak at wavelength about 500 nm which corresponds to the green luminescence. This is caused due to the vacancy defects in ZnO crystal structure. This high peak overshadows the 390 nm peak on PL curve.

9.4 Scanning Electron Microscope (SEM) Images:

SEM images of ZnO deposited polyimide substrates.

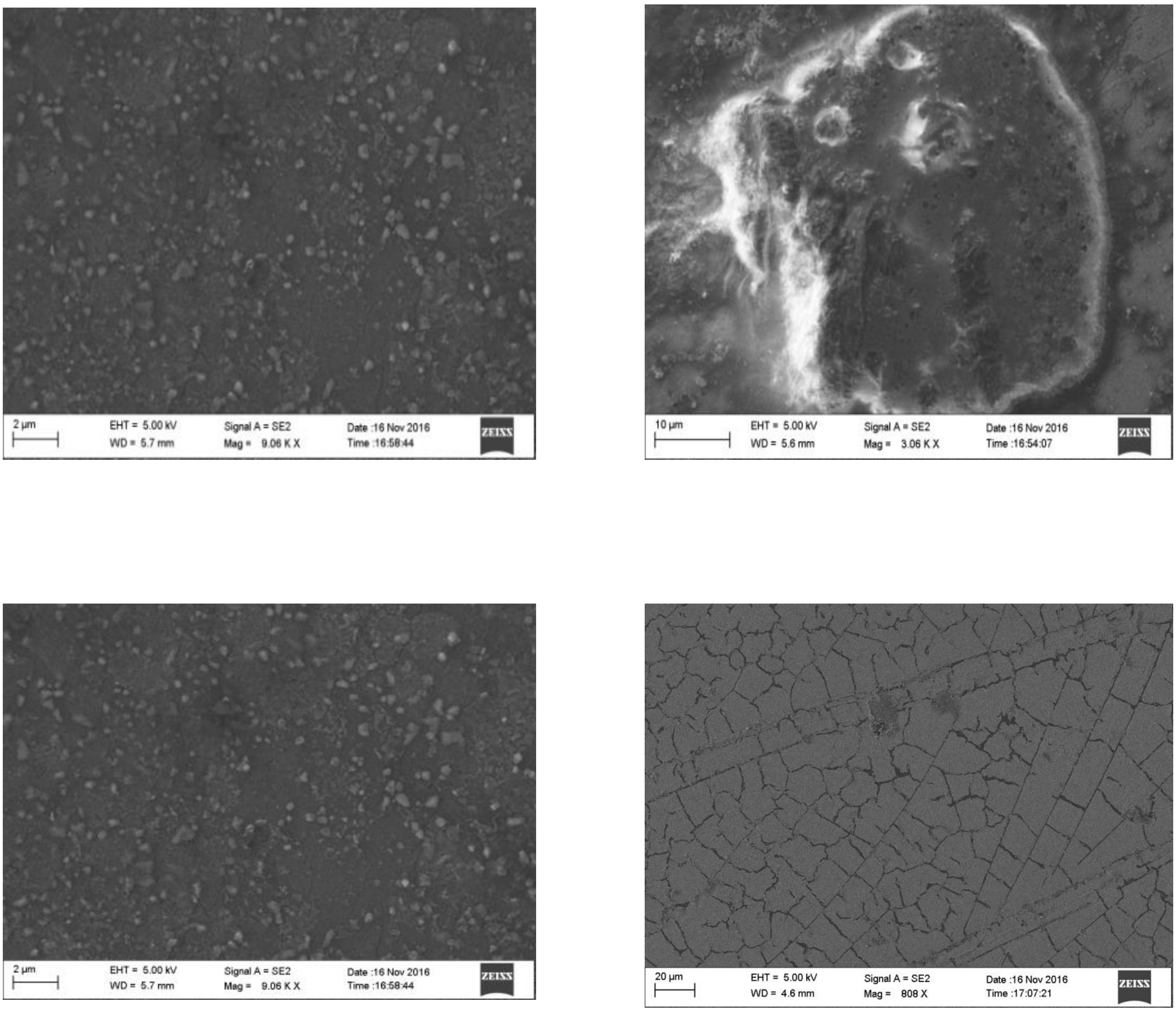

Fig. 20 – SEM IMAGES of ZnO on PI

Conclusion

Substrate treatment:

The surface pretreatment of polyimide sheet with KOH solution results in increased hydrophilic character of the substrate. The outcomes are satisfactory to some extent but on leaving substrate to the environment for a longer period of time the print quality gets degraded.

ZnO deposition:

ZnO ink printed polyimide has also shown satisfactory results. Further experiments can be done by varying ZnO concentration and also new inks can be synthesized using copper for better conductivity.

The component Hexylamine used in this entire experiment should be handled with caution. Also, other compounds which can be used as dispersing agent for Zinc salts should be studied and experiments can be done with them.

Ink thus formed reacts with rubber tubes of the inkjet printer which weaken the tubes and increases their brittle character. This damage of tube can be checked by either changing dispersing agents or by changing the material of the tubes.

Inkjet printing has proved to be a successful, easy and efficient method for printing even materials other than ink. Almost any kind of ink may it be conducting, insulating, resistive or dielectric can be synthesized and viscosity can be adjusted for printing by this method. Hence, there is a wide scope of further developing this cost saving and accurate method of printing electronic devices.

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