INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

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Project Report

Adhesion Enhancement of Conductive Inks on Non-porous Substrates for Printing Application

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Abstract

We explored the idea to enhance adhesion between the hydrophobic, inert surfaces of non-porous substrates and the deposited electrically conducting inks via an intermediate adhesive layer. The substrates considered included both glass and flexible counterparts like Polyethylene Terephthalate(PET) and Polyimide(PI). To pack more transistors and circuit elements within a given area requires a reduction in the line width of the interconnects. This, however, has a setback. The wettability of the ink formulation affects the limit to which we can reduce the line width. And if we tamper with the wettability the adhesion strength stands compromised. Hence our motivation was to optimize between high adhesion and low wettability such that the line width of the interconnects can be further reduced.

We spin-coated polymers such as Polyethylene Glycol(PEG), Poly-vinyl Pyrrolidone (PVP) and Polyacrylic Acid(PAA) in water and ethanol for the intermediate layer. Drying of these layers was carried out at 150*C. Further, a layer of silver ink was deposited by spin-coating to provide conductivity. Scotch Tape (3M Tape) test revealed that the adhesion strength improved with the intermediate layer. However, the wettability was also enhanced. optimizing the two features still remains.

Acknowledgements

I am deeply indebted to my project supervisors, Dr. Ashish Gupta and Prof. Anshu Gaur for their valuable guidance, fruitful discussion, constant support and encouragement throughout my SURGE program and for inculcating in me the spirit of scientific research. I am also grateful to Ms. Aruna, Mr. Piyush and Ms. Karishma for helping me with the experimentation and characterisation of the substrates prepared. Without their guidance, the project would not have come to fruition.

Introduction

Flexible Electronics is a growing field with tremendous potential. It has applications in Flexible Displays and Circuits, RFID Tags, Low-Cost Sensors, Organic Thin Film Transistors, Solar panels (bus bars and interconnects) and other disposable electronic devices. The fabrication of these devices could be carried out in two ways: Printing or Lithography. If the resolution lies in the feasible domain printing is preferred over the high cost associated with photo-lithography. The process is simple and accurate with high speed and reasonable resolution. It has low materials consumption and lesser wastage as compared to its counterpart.

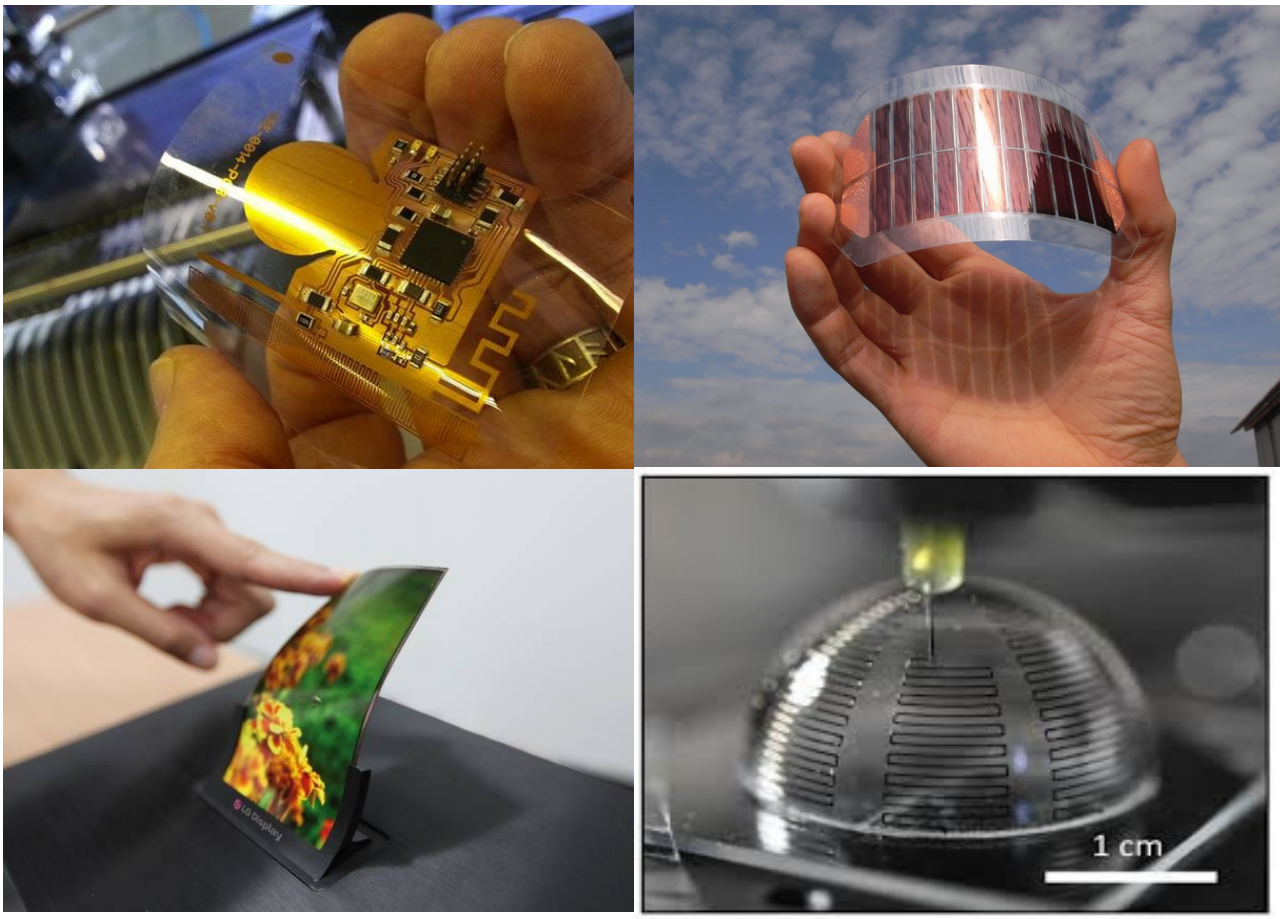


Figure 1. Areas of Application: (Anti-clockwise, from Top Left) Disposable electronic circuits, Solar panels, Antennas, Flexible Displays.

A. Printing Techniques

Four most used printing processes are Screen, Inkjet, Gravure, and Spray. The ink formulation for these processes is engineered depending on the viscosity and the surface tension coherent with it. Now to facilitate flexibility and high conductivity in the above-mentioned devices, we require polymer substrates and metal nanoparticles based inks (or MOD inks; metal-organic decomposition). Polymers by themselves do not show enough conductivity for practical significance. Besides, they are yet to contest the magnitude offered by patterns formed after sintering of metal nanoparticles.

These metal nanoparticles suspended in the ink formulation have a two-fold advantage. Due to their high surface energy, they have a low sintering temperature which makes way for short process time.

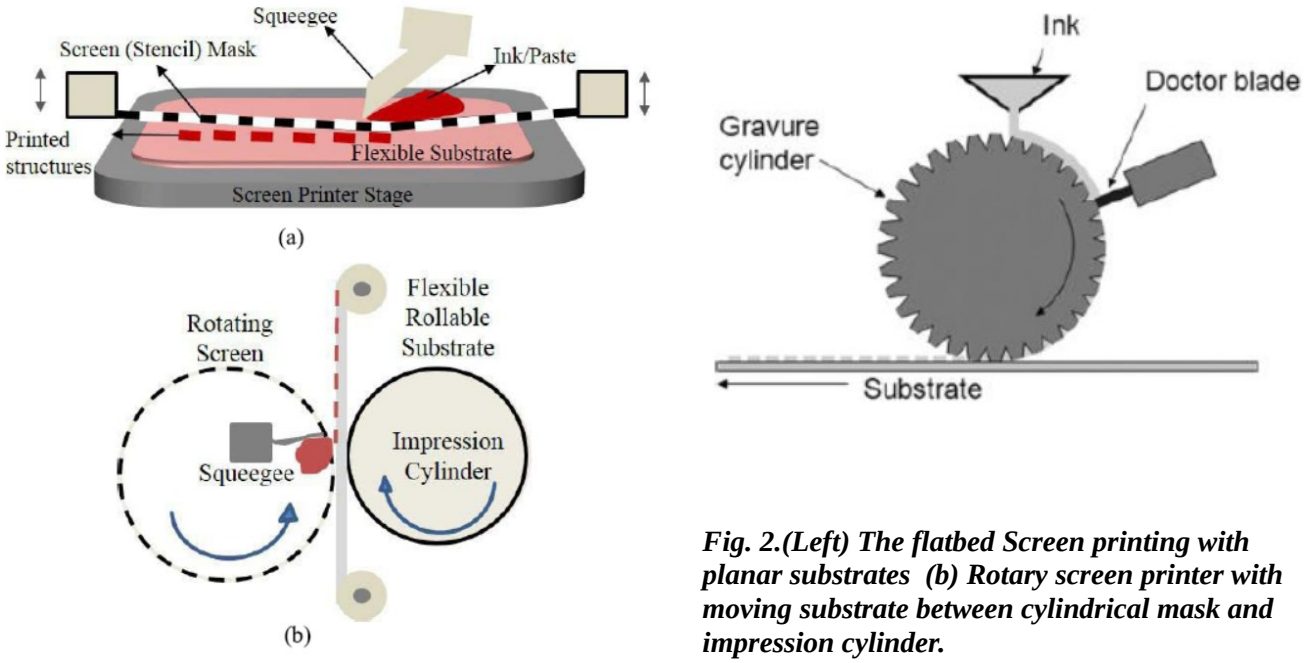


Fig. 2.(Left) The flatbed Screen printing with planar substrates (b) Rotary screen printer with moving substrate between cylindrical mask and impression cylinder.
(Right) Schematic of a gravure printing system.

time. This attribute of nanoparticles also permits deposition on flexible polymer substrates which have low glass transition temperature (Tg). Secondly, their dimension allows for higher resolution of the printed patterns. Silver, copper, and gold are among the potential candidates which chosen for precursors. The rest of the ink comprises a solvent, stabilizing agent/capping agent, binder, and reducing agent. Together, these components determine the adhesion, sintering temperature, printing method, conductivity and the resolution of the printed features.

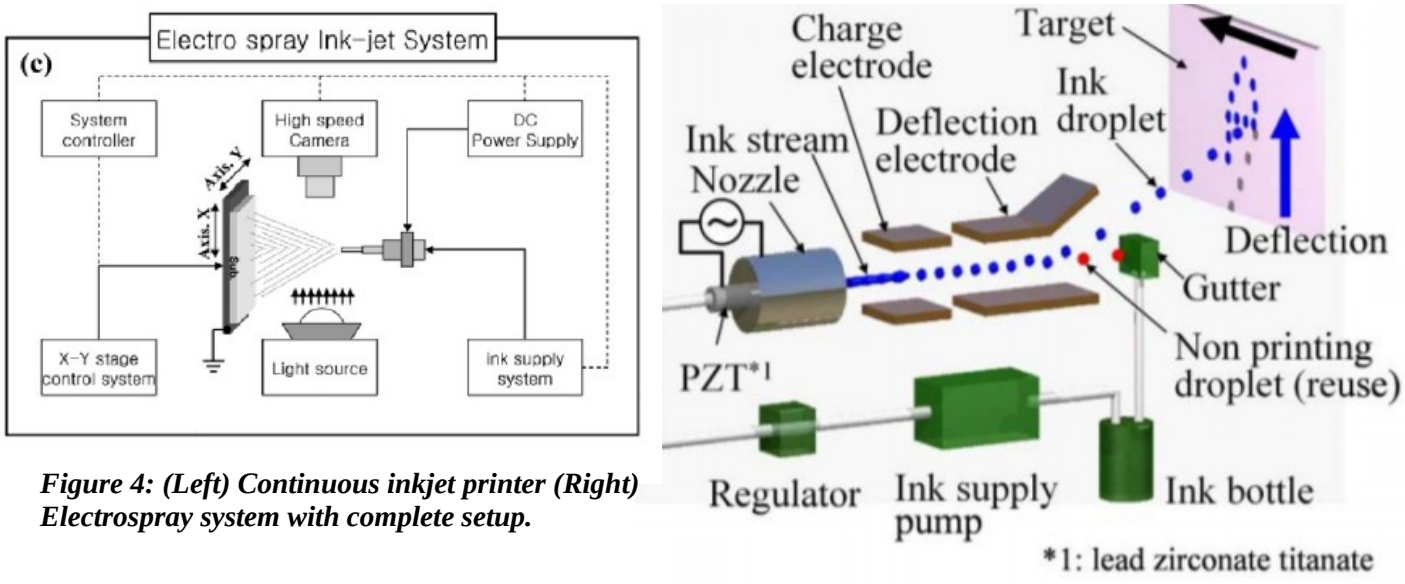


Figure 4: (Left) Continuous inkjet printer (Right) Electrospray system with complete setup.

B. Objective

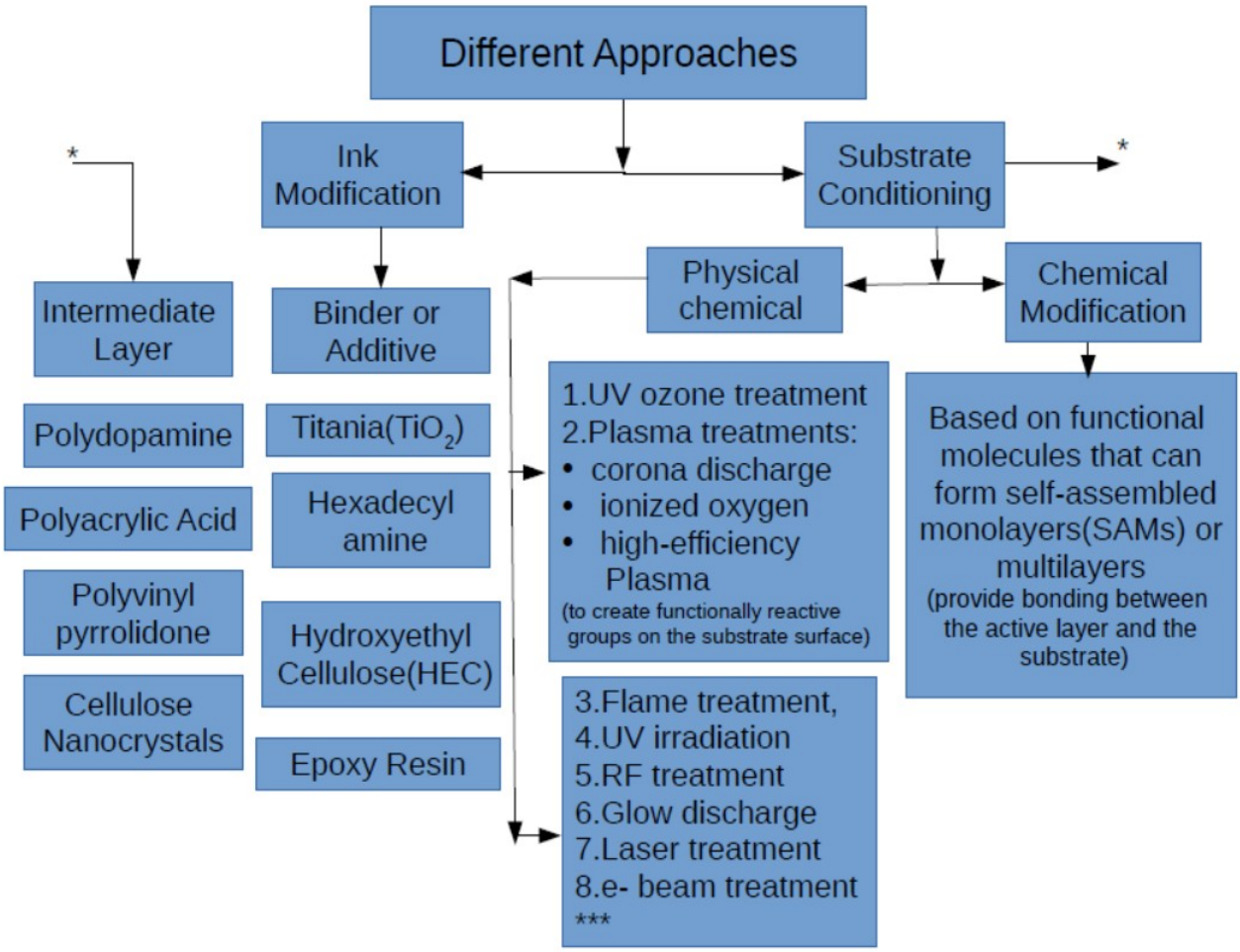
As envisaged by Thomas Moore, with time we've witnessed an increasing density of transistors integrated within a given area. And to ensure the same for flexible electronics, researchers are trying to decrease the line width of the printed patterns without compromising other characteristics. One of the crucial features that get affected when we reduce the line width is adhesion of the ink with the substrate. It is decisive for the integrity of the deposited circuit. Maintaining it requires enhanced interaction with the substrate. But wait, it's not as straightforward. If the cohesive forces are not strong enough, the high wettability of the ink with the surface would lead to spreading of the ink. Consequently, the line width would increase.

To tackle this problem, several approaches have been identified which can be broadly divided into two categories: ink modification and substrate conditioning. The motivation of this project was to improve adhesion by deploying an intermediate layer in between the metal nanoparticle ink and the substrate. We tried three different polymers in our experiments: Polyacrylic Acid (PAA), Poly-vinyl Pyrrolidone (PVP), Polyethylene Glycol (PEG). Low molecular weight powders of these polymers were taken at different concentrations in water and ethanol. After magnetic stirring for complete solubility, these solutions were spin-coated on three different non-porous substrates: Glass, Polyethylene Terephthalate (PET) and Polyimide (PI). Finally, two different silver ink formulations were deposited on these coated substrates. characterization results were analzsed and the adhesion magnitude was found to have increased.

Literature Review

A. Different Approaches

Papers in journals and patents suggested many ways to go about solving this problem. Largely they can be grouped under Ink Modification and Substrate Conditioning. Substrate conditioning could be achieved through physical treatments like UV-Ozone, RF, Plasma, Laser, etc. or through the costing of an intermediate layer. This intermediate layer provides bonding between the active layer(comprising the metal precursor) and the substrate. Ink modification, on the other hand, involves tweaking the components of the ink. Here, while the metal precursor, solvent, capping/stabilizing agent and the reductant are fixed, only the binder remains for modification.



These methods have their share of merits and demerits. From the industry point of view, some of their distinct disadvantages are:

(i) The addition of binders or additives to the ink results in a compromise with the conductivity of the printed features. Their presence in between the sintered nanoparticles obstructs the formation of a smooth conductive channel for the electrons to travel. For eg: 2% Hydroxyethyl Cellulose (HEC) and epoxy binder have been used for promoting adhesion.
Another issue which we face with binders is their high sintering temperature. Since some of them depend on inter-diffusion into the substrate for adhesion enhancement, they require relatively high temperatures for this process. In many cases, these temperatures are not compatible with substrates having low glass transition temperature.

(ii)Pre-treatment procedure like UV-Ozone, RF, Laser, Electron beam or Plasma treatments are both expensive and add an extra step to the fabrication process. Consequently, these steps are accommodated only in the absence of an alternative.

For these reasons, the substrate conditioning method of coating an intermediate layer is preferred over the others. A closer look into the plausible adhesion mechanisms gives us an idea of the ways in which we can influence this property.

B. Important Deductions

A common phenomenon was observed in papers which discussed the intermediate layer approach. The presence of an atom willing to share its lone pair of electrons. Silver and copper have a great affinity towards carbonyl bonds, sulfur and nitrogen atoms. And they were exploited for initiating bond formation with the active layer. The other end of the compound/chain interacted with the substrate. For eg: Self-assembled monolayers of silane coupling agents such as (3-mercaptopropyl)-trimethoxysilane (MPTS) and (3-aminopropyl)-trimethoxysilane (APTS) have been used earlier. However, they required plasma treatment of the surface for its activation to form alcohol bonds from, say the SiO_2 bonds present in glass.

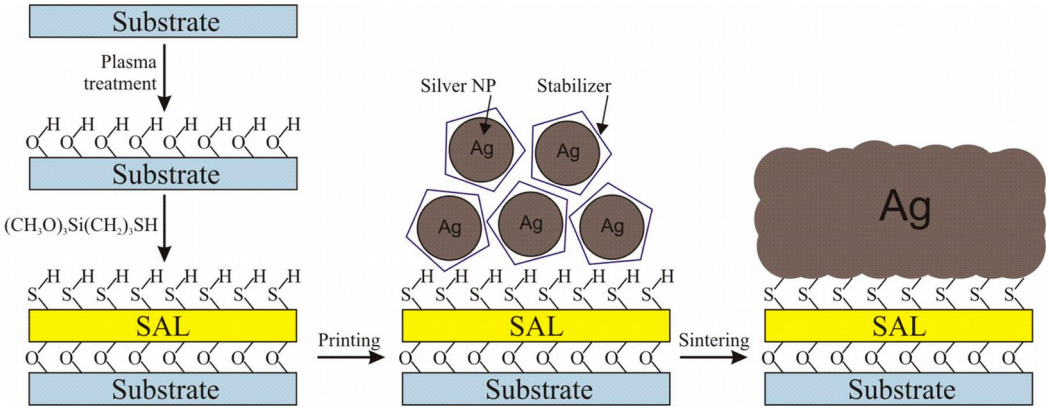


Fig 4. Scheme: (i) polymer substrate modification by plasma treatment, (ii) silanization by MPTS with the formation of SAL, (iii) printing of Ag ink, and (iv) curing of the printed ink.

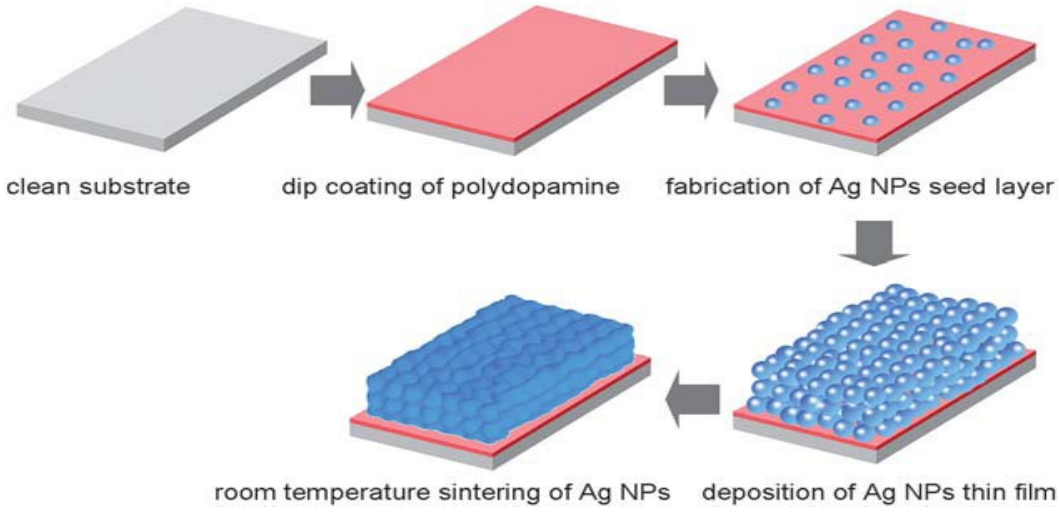


Fig. 1 A schematic illustration: The substrate is first modified by the dip-coating of polydopamine, followed by fabricating a Ag NPs seed layer on it. Afterwards, a silver-plating process is carried out on the modified substrate. By immersion in an electrolyte solution at room temperature, the Ag NPs coalesce and form a conductive thin film

C. Optimal Ink Design requirements include:

(i) Synthesis procedure should be both simple and high-yielding.
(ii)Ink should possess low viscosity to make it compatible with a broad range of patterning techniques
(iii) Patterned features should be highly conductive at room temperature and achieve bulk conductivity upon annealing at mild temperatures (<100 °C).
(iv) Finally, the ink should remain stable at room temperature for months without particle precipitation

D. Adhesion mechanism Discussion

Sungchul Ju et al. believe that the adhesion of silver nanoparticles could be mainly attributed to Van Der Waals forces of attraction. Determining factors for this type of adhesion include substrate hardness and adhesion distance. In general, adhesion level of silver nanoparticles increases as the substrate hardness decreases. Chemical bonding follows in the order of magnitude of adhesion strength and hence importance. These are followed by surface roughness effects. Surfaces with irregularities such as pores, holes, and crevices allow adhesives to penetrate into them. Consequently, the adhesive locks mechanically into the adhered surface.

Diffusion bonding is another crucial phenomenon which acquires significance at applied pressure and elevated temperatures. Here, the atoms of the adhesive must be compatible with the substrate in terms of diffusion and miscibility for inter-diffusion to take place.

Adhesion Mechanism	Adhesion Factor	Basic Adhesion Theory
Van der Waals force (P)	Substrate hardness Adhesion distance	Particle adhesion
Van der Waals force (A)	Surface energy change	Adhesive adhesion
Chemical Bonding	Bond species and density	Particle adhesion Adhesive adhesion
Mechanical Interlocking	Surface roughness	Adhesive adhesion
Silver to metal diffusion	Temperature	Particle adhesion

Table 1. Adhesion mechanisms and their determining factors

Procedure

A. Experimentation Steps

1. Substrate preparation

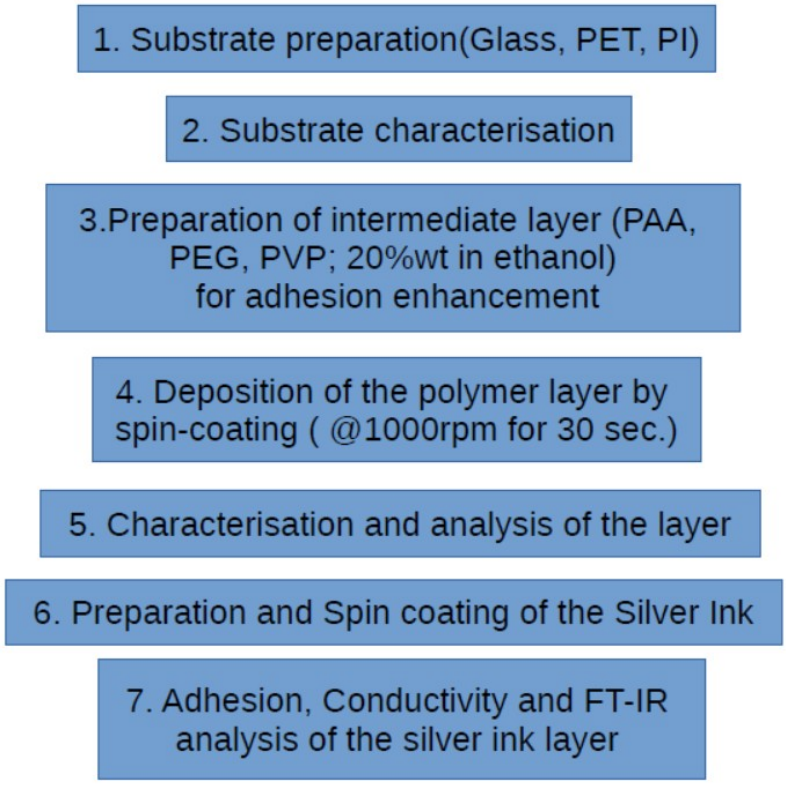
- Cut 1”x 1” sections of Glass, PET, PI
- Steps involved:
  (i) Sonicated them with IPA for 5 min followed by DI Water.
  (ii) Dried with N_2 air and subsequently with a vacuum pump.
- Cleaned with tissue paper

2. Substrate characterization

- Measured the contact angle with Goniometer
  => Three different solvents were considered for the measurement: DI Water, Isopropyl Alcohol (Polar) and Toluene (nonpolar)
- Calculated the Surface Energy of the substrates from the Contact Angle Data
  => The calculation was based on the Owens-Wendt formula:

$$(1+\cos(\theta))\gamma_{LV} = 2\sqrt{(\gamma_{SD}\gamma_{LD})} + 2\sqrt{(\gamma_{SP}\gamma_{LP})}$$

- Conducted FT-IR analysis to understand the chemical composition of the surface.



Flow Chart 1. Stepwise flow of the experimentation procedure

3. Preparation of intermediate layer for adhesion enhancement

- Selected the following polymers having low molecular weight:
  => PAA, PEG (1500), PVP (MW= 40,000)
- Steps involved:
  (i) Took 1gm of the polymer (preference- lower MW) and added it to 9ml DI water (conc. of solution 10wt%)

(ii) Magnetic stirring for 3 hrs at room temperature.
(iii) Repeated preparation of the polymer solution with higher concentration when 10%wt did not coat uniformly. Increased it to a maximum of 20wt%.
(iv) Repeated the experiment with ethanol as the solvent to compare which one gave better results.

4. Deposition of the polymer layer and drying

- Spin coated the three polymer layers on different substrates with the following parameters: ~ 1000 rpm for 30 sec and acceleration time = 10 sec
- Dried the substrates in a furnace @ 150*C for 2 hours.

5. Analysis after layer Deposition

- Contact Angle measurement with Goniometer
- Surface energy calculation based on contact angle data
- FT-IR analysis to understand the chemical composition of the surface and observe the changes in the spectrum revealing the formation of new bonds

6. Deposition of Silver Ink

- Spin coated two different Silver ink formulations @ 1000rpm for 30 sec
  =>Ag I-49 and Ag I-50.
- After deposition of silver ink, the substrates were heated in the furnace @ 150*C for 2 hours for initiating sintering of nanoparticles.

7. characterization of the Silver Ink

- Adhesion strength through Scotch Tape Test;
- ***Four-probe method for conductivity analysis of the pattern printed (Still remains)***
- Conductivity with a digital Multimeter.
- FT-IR analysis to understand the chemical composition of the surface and ascertain the mechanism behind enhanced adhesion.

B. characterization Techniques

After the deposition/coating of a conducting layer, it is analyzed by several techniques which help us characterize it. The results obtained are co-related to find out the underlying mechanism operating behind adhesion enhancement.
For eg: XPS or FT-IR spectroscopy provides us information about the surface chemical composition which tells us whether there any new chemical bonds have formed or not. The size and shape of the Ag NPs, on the other hand, gives us an idea of the sintering temperature and the obtained conductivity of the printed patterns.

Characterization Parameter	Instrument
1. Size and shape of the synthesized Ag NP and the microstructures	Scanning electron microscopy
2. Crystal and chemical structure	X-ray diffractometer and XPS
3. Surface charge	Zeta-potential analyzer
4.Rheological behavior	Modular compact rheometer
5. Thermal decomposition behavior	Thermal gravimetric Analysis, DSC
6. Morphology	Surface profiler
7. Resistivity	Four-point probe station
8. Adhesion	Scotch Tape Test
9. Chemical composition of the surface	Fourier Transform Infra-red Spectroscopy

Table 2. Important Characterisation Parameters and the corresponding instruments to obtain them.

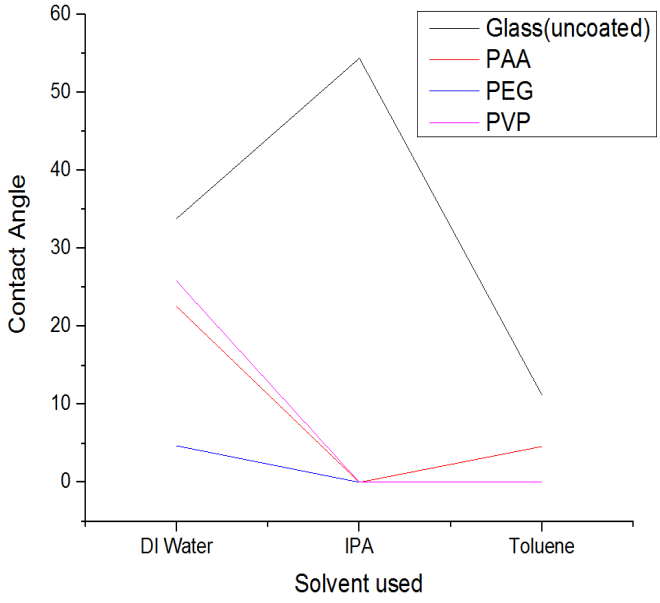
Results and Discussion

A. Characterization Results

a) Contact Angle

=> The instrument used was Dataphysics OCA 15EC Goniometer.
=> Contact angle measurements for all three substrates (coated and uncoated) were carried out with different solvents (polar, nonpolar and DI Water). This was done to find out the change in wettability of distinct types of solvents upon surface modification of the substrate.

i. Glass



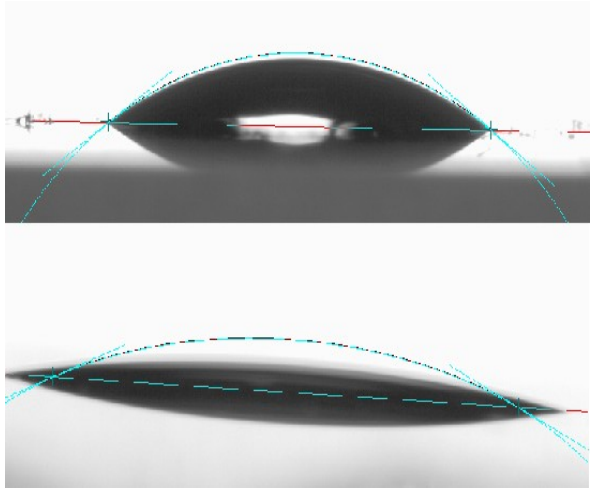
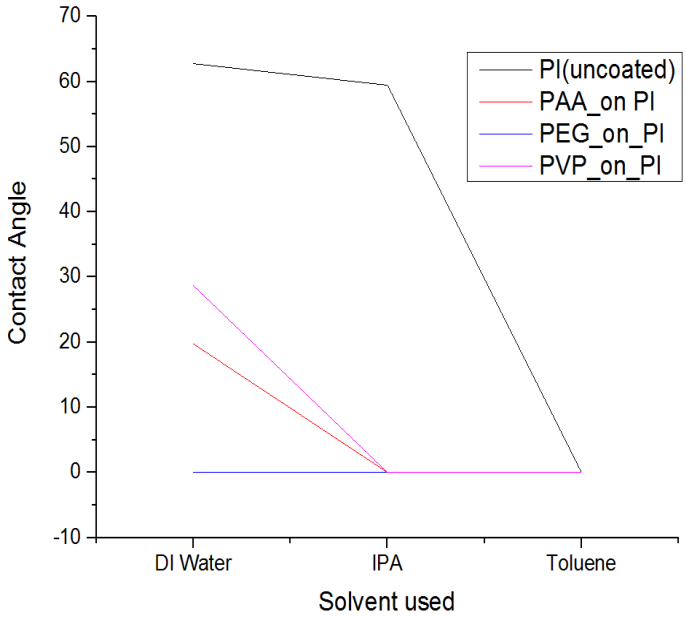


Figure 5. (Left) Contact angle measurements for plain and modified glass substrate. (Right) Increased wettability of DI Water after coating with PVP.

ii. Polyethylene Terephthalate (PET)



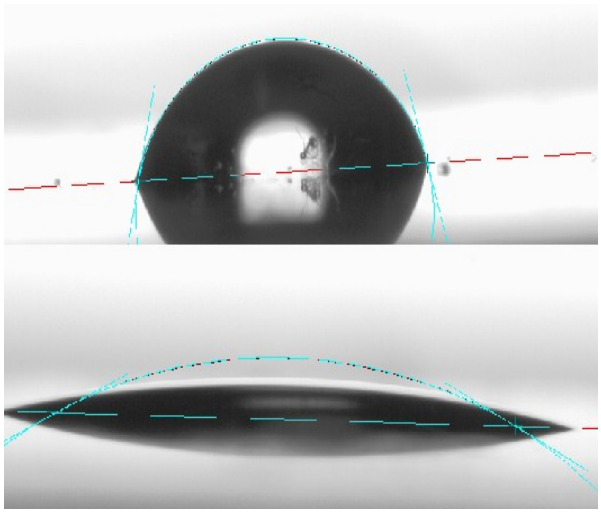
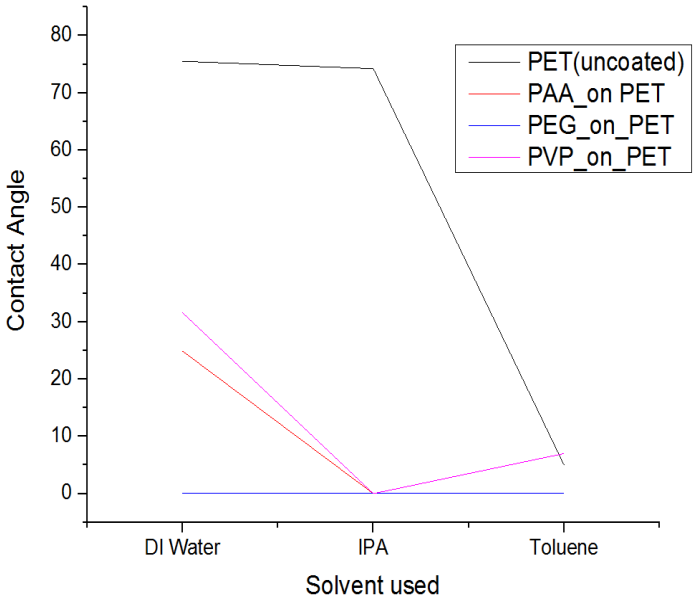


Figure 5. (Left) Contact angle for plain and modified PET substrate. (Right) Increased wettability of DI Water after coating with PVP.

iii. Polyimide (PI)



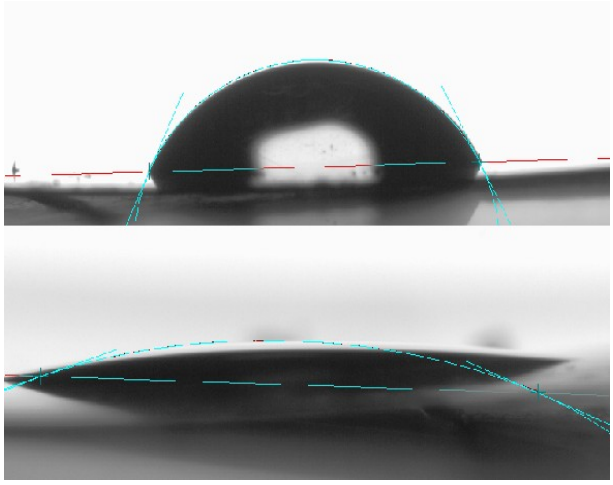


Figure 5. (Left) Contact angle for plain and modified PI substrate. (Right) Increased wettability of DI Water after coating with PVP.

Observations:

- Upon modification of the substrates with the intermediate polymer layers, the wettability increased indicating a high surface energy. One reason could be the compatibility with the solvent i.e. ethanol, comprising both polar and nonpolar parts.
- Change in the contact angle was observed to be minimum in the case of Toluene. One reason for this could be the low compatibility with Ethanol (solvent of the intermediate layer) because of the small nonpolar component in the molecule. With DI Water and IPA, however, a significant change in the wettability was observed.

b) Surface Energy

=> This characteristic was calculated by the Owens-Wendt formula, according to which:

$$(1+ \cos(\theta))\gamma_{LV} = 2\sqrt{(\gamma_{SD}\gamma_{LD})} + 2\sqrt{(\gamma_{SP}\gamma_{LP})}$$

Here,

- θ = Contact Angle
- γ = Surface Energy
- S, L, V = Solid, Liquid, Vapour
- D, P = Dispersive, Polar

The values for the uncoated substrates were (in milli-Newtons per meter) :

i. Glass - 64.84 mN/m
ii. PET - 44.44 mN/m
iii. PI - 27.42 mN/m

Observations:

- The surface energy data for the coated substrates could not be obtained because of high wettability of the solvents. In some cases, it was as low as zero degrees.
- Clearly, the high wettability of the coated substrates implies high surface energy.
- Here, optimizing between the wettability and adhesion remains.

c) Fourier Transform Infra Red Spectroscopy (FT-IR)

=> The instrument used here was a Bruker Vertex 70 FT-IR spectrometer.
=>The transmittance spectrum of the substrates were analyzed to understand the chemical composition of the surface.

i. Plain Glass

Functional Group	Frequency(cm-1)
Alcohol OH stretch	3600-3200
Carboxylic acid OH stretch	3600-2500
C=O aldehyde	1740-1720
C=O ester	1750-1720
C=O ketone	1745-1715
C=O amide	1700-1500
C=C aromatic	1600-1400
C-O-C stretch	1250-1050 several
C-OH stretch	1200-1020

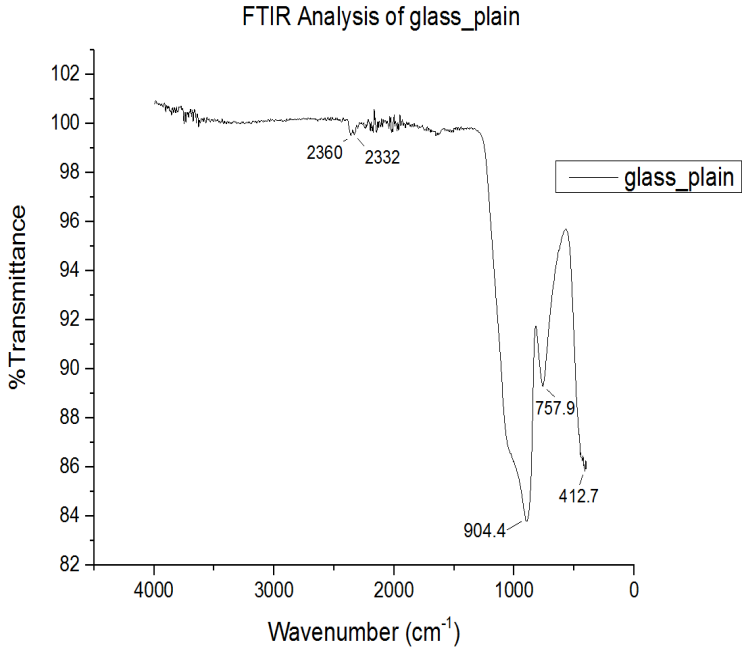
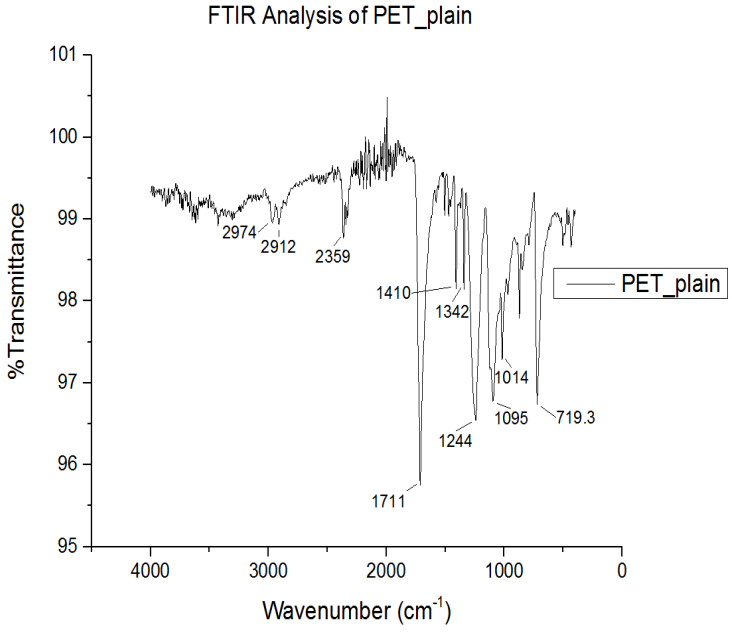


Fig 6.(Left) IR Bond Table for reference. (Right) FT-IR spectrum of Plain Glass

ii. Plain PET

iii. Plain PI



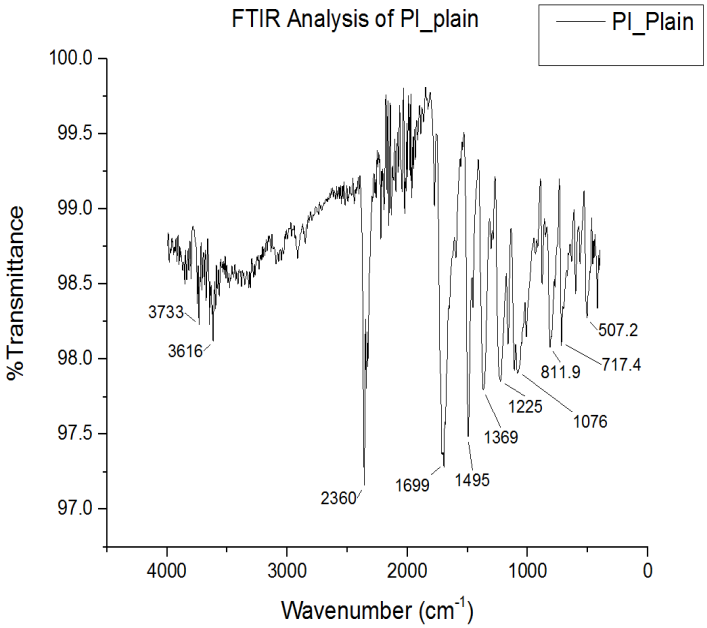
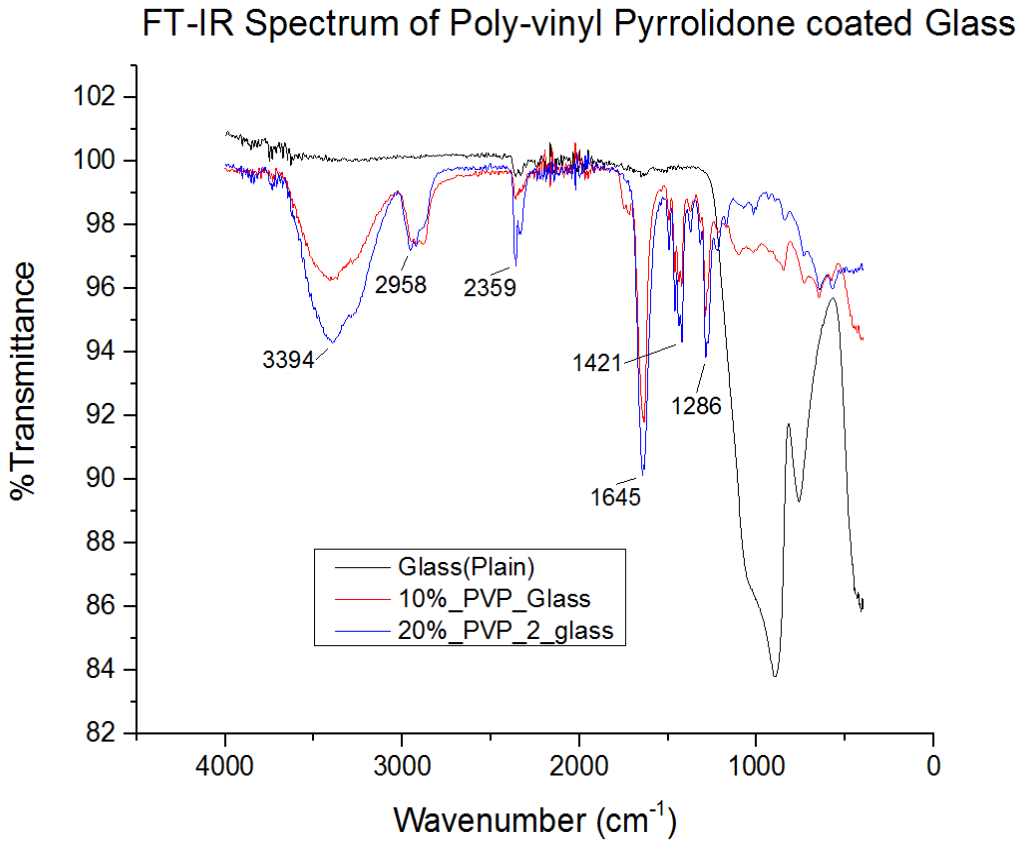
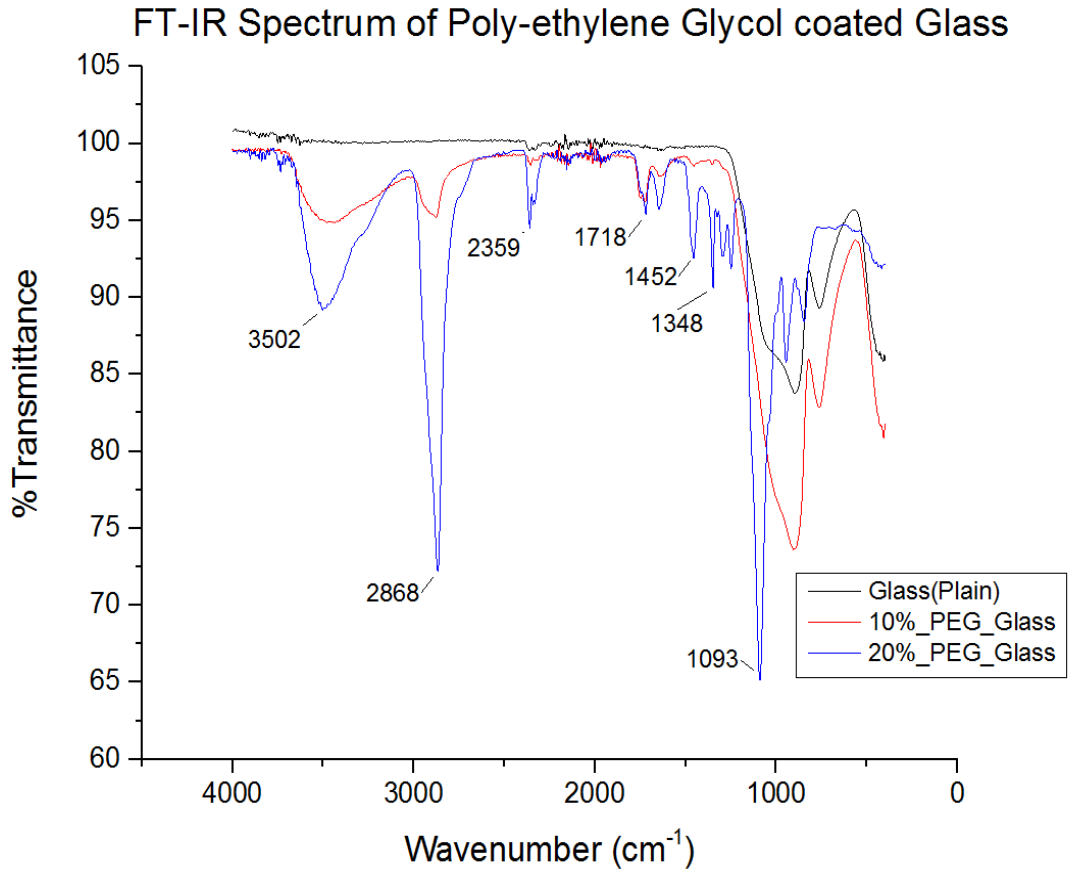


Fig 7.(Left) FT-IR spectrum of Plain PET (Right) FT-IR spectrum of Plain PI

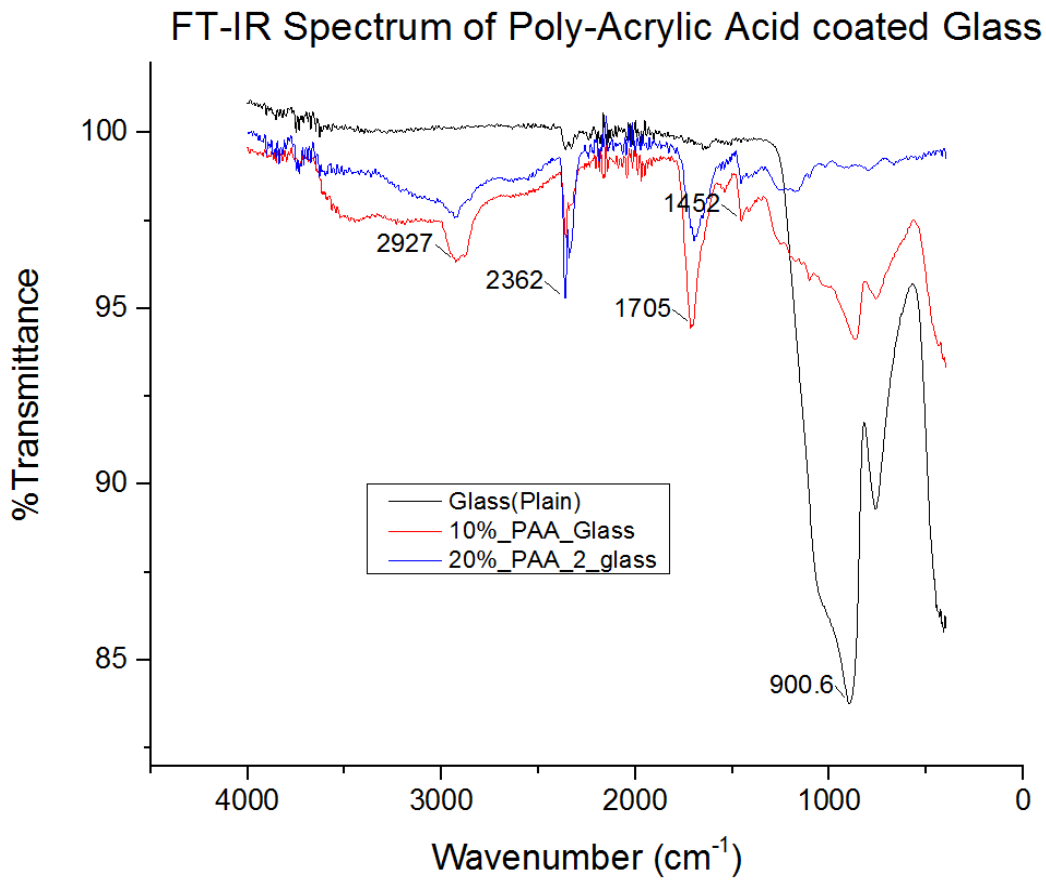
iv. PVP coated Glass



v. PEG coated Glass



vi. PAA coated Glass



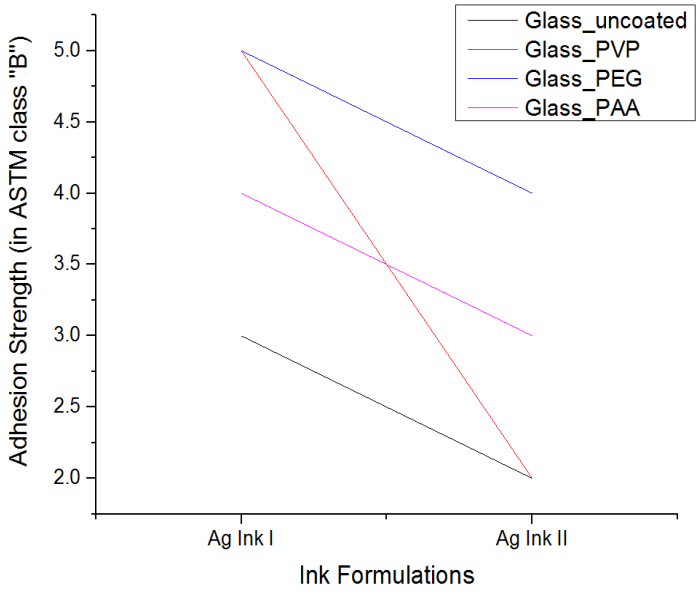
Discussion

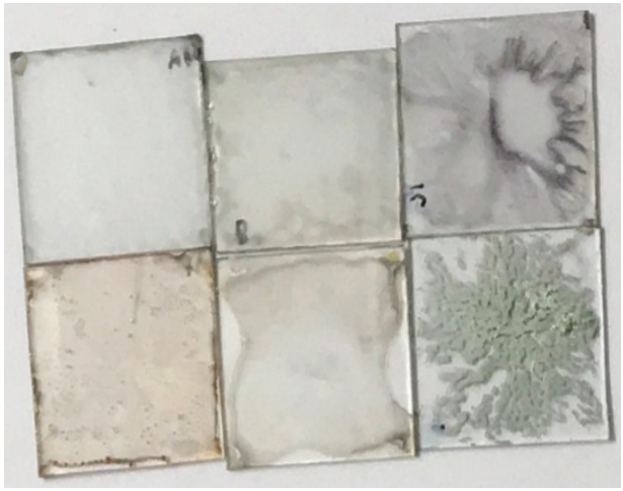
- The FT-IR results indicated the formation of an even and uniform film in the case of PAA and PVP. This was confirmed by obtaining a verified graph of the coated substrate and comparing the two. However, PEG deposition was both patchy and non-uniform, which is confirmed by 1093 cm-1 peak.
- To confirm whether any bond formation took place in between the substrate and the intermediate layer, the FT-IR graphs have to be analyzed further. Besides, we need to try sintering at different temperatures and see if bond formation changes and/or adhesion improves.
- With increasing concentration of the polymer, the peak intensity increased indicating better coating on the respective substrate.

d) Adhesion Strength of Coating

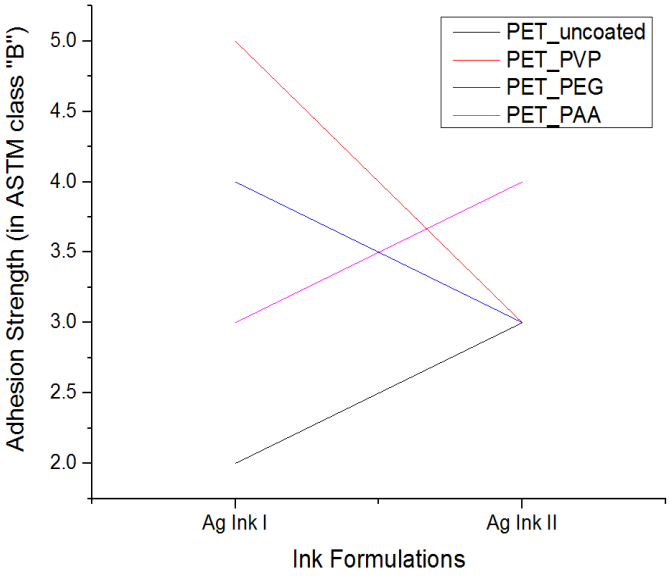
=> The Tape considered for the Peel Test was the 'P' grade adhesive tape of industrial standards.
=> According to the ASTM standards the degree of adhesion is decided by the % of the area that gets affected after the peel off test. (5B => above 65% and 0B => no change)

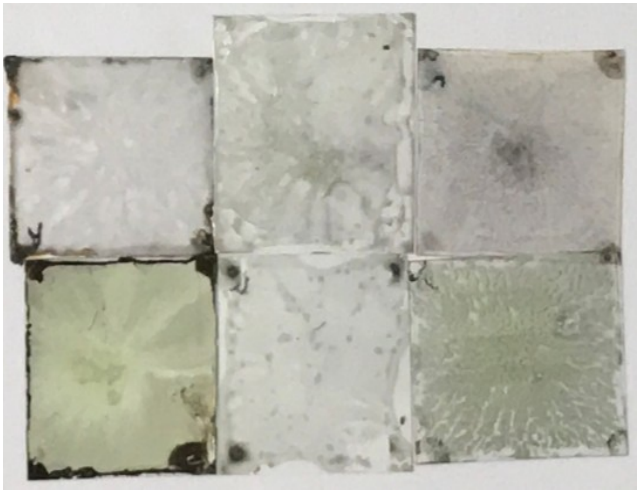
i. Glass





ii. PET





Observations:

- Adhesion strength enhanced in all cases of coated substrates compared to the uncoated ones.
- What still remains for examination is the surface profile and film thickness after the coating.
- These results may not be accurate enough since the silver coating was neither uniform nor clearly visible. Further characterization of the film would provide better insight.

e) Conductivity

- Conductivity measurements were carried out with a multimeter (Rish 18S).
- Neither of the substrates (coated or uncoated) showed any conductivity, whatsoever. Spin-coating more layers of the ink should makeup or dip-coating could be a better alternative.
- Another alternative that we could consider is a wire-bar coating which provides an assured thick film of the ink. The functioning of the process is similar to Screen Printing.
- We could try measuring this characteristic with a four probe station to find if the silver film formed has a resistivity of the order of nano ohm-m.

Conclusion

We explored three polymers (PAA, PVP, PEG) for adhesion enhancement of conductive metal nanoparticle inks (with silver based metal precursor) via an intermediate layer. Non-porous substrates like glass, PET and PI were considered for spin-coating of this layer. The powder form of these polymers was dissolved in water and ethanol at different concentrations; starting from 5wt% to 20wt%. After drying these layers in a furnace, different silver ink formulations were spin-coated and sintered at 160*C for 2 hours. Though the adhesion of the coated film was enhanced, the coating of the film needs to be scrutinized further to ascertain is integrity. We also observed that the wettability of solvents (DI Water, Isopropyl Alcohol, and Toluene) improved for all three polymer intermediate layers.

The motivation of the project was to be able to reduce the line width of the conductive patterns deposited by printing techniques. Here, it was crucial that we achieved the objective through an intermediate layer without increasing the wettability tremendously. It was important to control and regulate the wettability so that the ink doesn't spread much. This would affect the line width. Hence we wanted to optimize between high adhesion and low wettability such that the line width of the interconnects could be further reduced. In the limited time period, however, we could not achieve it.

Scope for Future Work

The intermediate layer formed in the experiments was processed at a fixed concentration, sintering temperature, and spin-coating parameters. We will try tweaking these conditions later to further improve results. Different deposition/coating/printing techniques such as screen printing or wire-bar drawing could be tested to obtain higher adhesion and conductivity of high-resolution features.

From the literature review, we had short-listed two other potential candidates which could be deployed in the intermediate layer. They were polydopamine and Cellulose nanocrystals. Poly-dopamine, a mussels inspired polymer has been claimed to provide promising adhesion according to Yuhua Long et al.

In the future, we will also try analyzing the surface profile and film thickness. This would assure us of the integrity of the silver film. It would make the analysis more rigorous.

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